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MICROWAVE REGENERABLE AIR PURIFICATION DEVICE

James E. Atwater
John T. Holtsnider
Richard R. Wheeler, Jr.

August 1996

FINAL REPORT

CONTRACT NAS2-14374

Prepared for:

NASA AMES RESEARCH CENTER
MOFFETT FIELD, CALIFORNIA

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URC 80647

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TABLE OF CONTENTS

PROJECT SUMMARY 1

I. INTRODUCTION 2

II EXPERIMENTAL SECTION 4
Experimental Approach 4
Sorption-Desorption Studies 4
Microwave Spectral Studies 4
Materials and Apparatus 5
Microwave Powered Thermal Desporption Apparatus 5
Multifrequency Microwave Transmission and Reflection Apparatus 9
Sorbernts 11
Contaminants 11
Analytical Instruments 11

III. RESULTS AND DISCUSSION 12
Microwave Powered Thermal Regeneration of Sorbents 12
Sorption and Thermal Desorption of Acetone 13
Sorption and Thermal Desorption of Carbon Dioxide 16
Sorption and Thermal Desorption of Trichloroethylene (TCE) 27
Sorption and Thermal Desorption of Water Vapor 32
Sorption and Regeneration of Layered Media for CO_2, Water Vapor and Trace Organics 38
Microwave Reflection and Transmission Spectra 44

IV. CONCLUSIONS 61

V. REFERENCES 63

LIST OF FIGURES

1. Microwave Powered Thermal Regeneration Experimental Apparatus 6
2. Microwave Power versus Variable Transformer Setting 7
3. Device for Mounting Sorbent Beds in the Rectangular Waveguide 7
4. Sparging Apparatus for Humidifying the Contaminated Gas Stream 8
5. Multifrequency Microwave Transmission Test Apparatus 10
6. Sorption of Acetone on Activated Carbon 14
7. Thermal Regeneration of Acetone Loaded Activated Carbon 15
8. Sorption of Acetone on Carbosieve S-III 17
9. Thermal Regeneration of Acetone Loaded Carbosieve S-III 18
10. Sorption of Acetone on Zeolite ZSM-5 19
11. Thermal Regeneration of Acetone Loaded ZSM-5 20
12. Chemisorption of CO_2 by LiOH 22
13. Sorption of CO_2 by Molecular Sieve 5A 23
14. Microwave Powered Thermal Decomposition of Silver Carbonate 26
15. Sorption of CO_2 on Composite MS/5A 28
16. Thermal Regeneration of CO_2 Loaded MS 5A Composite Bed 29
17. Sorption of TCE on Activated Carbon 30
18. Regeneration of TCE Loaded Activated Carbon 31
19. Sorption of TCE on Carbosieve S-III 33
20. Regeneration of TCE Loaded Carbosieve S-III 34
21. Thermal Regeneration of TCE Loaded ZSM-5 35
22. Initial Sorption of Water Vapor on Silica Gel 36
23. Sorption of Water Vapor on Silica Gel Following Regeneration 37
24. Initial Sorption of Water Vapor on Molecular Sieve 13X 39
25. Sorption of Water Vapor on Molecular Sieve 13X Following Regeneration 40
26. Initial Sorption of Water Vapor on Molecular Sieve 5A 41
27. Thermal Regeneration of Water Loaded Molecular Sieve 5A 42
28. Sorption of Water Vapor on Molecular Sieve 5A Following Regeneration 43
29. Composite Bed: Initial Sorption of CO_2, Water Vapor, Acetone and TCE 45
30. Composite Bed: Repeat Sorption after Microwave Powered Regeneration 46
31. Relative Transmitted and Reflected Power for Empty Sample Chamber 47
32. Relative Transmitted and Reflected Power: CECA-8301MC Activated Carbon 48
33. Relative Transmitted and Reflected Power: Alltech 5769 Activated Carbon 49
34. Relative Transmitted and Reflected Power: Lithium Hydroxide 50
35. Relative Transmitted and Reflected Power: Molecular Sieve 5A 51
36. Relative Transmitted and Reflected Power: Molecular Sieve 13X 52
37. Relative Transmitted and Reflected Power: Silica Gel 53
38. Relative Transmitted and Reflected Power: Silica Gel with Adsorbed Water 54
39. Relative Transmitted and Reflected Power: Silver Carbonate 55

LIST OF FIGURES (Continued)

40. Transmitted Power Phase Shift:Top-Empty Cell, Bottom-CECA-830IMC 56
41. Transmitted Power Phase Shift:Top-5769 Activated Carbon, Bottom: LiOH 57
42. Transmitted Power Phase Shift: Top-MS 5A, Bottom-MS 13X 58
43. Transmitted Power Phase Shift: Top-Dry Silica Gel, Bottom-Wet Silica Gel 59
44. Transmitted Power Phase Shift: Silver Carbonate 60

LIST OF TABLES

I. Summary of Individual Sorption/Regeneration Experiments.. 12

II. Waveguide Microwave Heating Tests Results at 2.45 GHz 60

PROJECT SUMMARY

Feasibility of the use of microwave heating for the fast and efficient thermal regeneration of sorbents for the removal of carbon dioxide, water vapor, and trace organics from contaminated air streams has been conclusively demonstrated. The use of microwave power offers several advantages, including: improved heat transfer, lower thermal losses, improved power utilization, and enhanced operational capabilities.

During the Phase I research the sorption and microwave powered thermal desorption of acetone, trichloroethylene (TCE), carbon dioxide, and water vapor was studied at 2.45 GHz using a rectangular waveguide based test apparatus. Both activated carbon and Carbosieve S-III were identified as excellent microwave regenerable sorbents for use in the removal of airborne organics. Water loaded silica gel, Molecular Sieve 13X, and Molecular Sieve 5A were also effectively regenerated under microwave irradiation at this frequency. Molecular Sieve 5A and a carbogenic molecular sieve prepared at NASA's Jet Propulsion Laboratory were identified as viable microwave regenerable CO_2 sorbents. A sorbent bed containing multiple media was challenged with air containing 0.5% CO_2, 300 ppm acetone, 50 ppm TCE, and saturated with water vapor. The composite bed was shown to effectively purify the contaminated air stream and to be completely regenerated by microwave induced heating.

Spectral studies of the reflection, transmission, and phase shifts of microwaves for a variety of sorbents over the frequency range between 1.3 - 2.7 GHz have shown that the dielectric loss characteristics are strong functions of frequency and material. Frequencies have been identified with potential for more effective microwave heating of specific sorbents since these loss characteristics are responsible for microwave heating. Based upon these results, further development of this highly promising technology is highly recommended.

In addition to the obvious applicability to EVA and Advanced Life Support, two specific systems with strong potential for commercial application have also been identified. These are the acetone-Carbosieve S-III and TCE-ZSM-5 combinations. The first system represents an environmentally benign method for the recovery of waste solvents in a variety of industrial chemical processes. Using the highly selective carbon based molecular sieve, and the extremely rapid thermal desorption capabilities inherent to microwave heating, acetone (or similar solvents) can be recovered from waste gas streams by sorption and then concentrated by flash thermal desorption for collection by condensation. The second commercial application exploits the fact that ZSM-5 is not only a sorbent for removal of airborne trichloroethylene, it is also an effective catalyst for the deep oxidation of this contaminant, particularly in the chromium form. Thus, the TCE-ZSM-5 system forms the basis for a combined environmental remediation process to achieve both the separation and the ultimate destruction of TCE.

I. INTRODUCTION.

An investigation has been conducted to determine the feasibility of using microwave power to promote thermal regeneration of sorbents applicable to the life support requirements of astronauts during Extravehicular Activity (EVA). The primary advantages inherent to the use of microwaves are the increases in energy efficiency which result from the heating of materials by direct absorption of energy (in contrast to the indirect supply of heat by thermal conduction from heating elements), and the extremely rapid rates of heating which are possible. This maximizes the efficiency of energy transfer and minimizes conductive, convective, and radiative losses as well as the need to heat additional thermal mass[1]. These features suggest the possibility of a more compact and thermally efficient technology for the removal of CO_2, H_2O, and trace organic contaminants from air during EVA. A second generation thermally regenerable air purification system is envisioned in which microwave power is applied directly to the PLSS via coaxial cable, thus providing a means for regeneration which does not require removal of sorbent cartridges from the EMU. A third generation system could provide capability for sorbent regeneration during EVA. While the current investigation has been directed specifically toward EVA, the operational efficiencies which can be gained by the use of microwave power may apply equally to other life support requirements such as Air Revitalization within the cabin of spacecraft or within other enclosed space habitats.

Susceptibility to microwave heating is a function of the dielectric properties of the material. Microwaves encompass the upper end of the radio frequency (RF) electromagnetic spectrum. Due to their relatively long wavelengths, the behavior of microwaves is different in many respects from that of more energetic (and for most researchers more familiar) regions of the spectrum such as IR, visible, and UV light. If a high frequency RF signal is applied to a conductor, a current will flow. If the same signal is applied to a non-conductor (i.e. a dielectric material) then electromagnetic waves are propagated. If the electromagnetic energy is absorbed by the dielectric, the temperature of the material rises in proportion to the energy absorbed. The most common embodiment of microwave heating is the microwave oven in which 2.45 GHz microwaves couple primarily with the rotational transitions of dipolar water molecules. Different dipole-dipole loss phenomena occur in solids such as silicon carbide (SiC) and barium titanate ($BaTiO_3$) which couple very effectively with microwaves. In these materials the frictional losses which result in the generation of heat are manifested through oscillatorily induced polarizations (Maxwell-Wagner effect, etc.) rather than by rotationally induced internal friction[2,3].

In concept, microwave powered thermal regeneration of a loaded adsorbent can occur via three possible mechanisms: 1) the adsorbent only couples with the microwaves; 2) both adsorbent and sorbate couple; and 3) the sorbate only couples. From an energy efficiency perspective, the latter mechanism offers the greatest potential savings in comparison to conventional thermal regeneration methods. Similar mechanisms apply to sorbents such as lithium hydroxide (LiOH) and silver oxide (Ag_2O) in which the contaminant is chemically bound, i.e., as Li_2CO_3 and Ag_2CO_3, respectively. Regeneration of chemisorbed materials will, in general, require higher temperatures, longer regeneration times, or both. The ability of microwave heating to achieve extremely rapid heat-up to very high temperatures may be a particular advantage when applied to the regeneration of these sorbents, and may make possible the regeneration of sorbents which otherwise would not be practical.

For the purposes of the initial investigation, a representative variety of candidate sorbent materials which are potentially useful in the removal of airborne water vapor, CO_2, and trace organics were screened for susceptibility to thermal regeneration using microwave power. These experiments were conducted at 2.45 GHz. In addition, because the susceptibilities of materials to microwave heating vary with the frequency of incident radiation, a variety of sorbent materials were also screened for their dielectric loss characteristics over a range of frequencies between 1.37 - 2.6 GHz. In these experiments, bulk properties were measured, with contributions arising from both the sorbent and the gas phase filling the pores and intergranular spaces[4-9].

II. EXPERIMENTAL SECTION.

Experimental Approach. To explore the usefulness of microwave power for the thermal regeneration of sorbents, the Phase I research effort focused on two primary areas of experimentation: 1) characterization of the susceptibility of a typical range of sorbents loaded with common airborne contaminants toward microwave powered thermal regeneration using 2.45 GHz microwaves; and 2) investigation of the spectral responses of sorbents over a range of incident microwave frequencies to determine if particular regions of the microwave spectrum exist which may be especially well suited for use with a particular sorbent.

Sorption-Desorption Studies. A variety of sorbents were selected for study, including: activated carbon, carbon based molecular sieves, lithium hydroxide, silica gel, silver oxide, and zeolite molecular sieves. Acetone, carbon dioxide, trichloroethylene, and water vapor were selected as challenge contaminants. Small packed sorbent beds were exposed to humidified air streams containing the appropriate contaminants and the relationships between cumulative flow and breakthrough of the sorbates were monitored. Once breakthrough occurred, the sorbents were exposed to microwave energy and the bed temperatures and effluent gas concentrations were monitored. For the cases in which successful thermal regeneration was indicated, this was confirmed by a subsequent re-loading of the sorbent with the contaminant to ensure that substantial sorption capacity had been regained.

Microwave Spectral Studies. Low power studies were conducted to examine the dielectric loss characteristics of a selection of typical sorbent materials over a range of frequencies. The dielectric loss properties determine a material's susceptibility to microwave heating. These properties are functions of both frequency of the incident microwave radiation and of the temperature of the medium. Owing to the time and monetary constraints inherent to the Phase I effort, room temperature experiments were conducted over the relatively narrow range of frequencies between 1.3 - 2.7 GHz. A vector network analyzer based system was assembled to monitor three properties of each test specimen: relative reflected power, relative transmitted power, and the phase shift of the transmitted power.

Materials and Apparatus.

Microwave Powered Thermal Desorption Apparatus. The microwave transmission and irradiation testbed is illustrated in Figure 1. The apparatus is composed of a series of WR 430 rectangular waveguide elements, and includes a magnetron, shorting plate, waveguides, directional coupler, solid state microwave detector, RF power meter, and a water load. An 800 W magnetron, emitting at 2.45 GHz, is located in the launcher waveguide section. The launcher section is terminated at one end by a shorting plate located at a distance of ¼ wavelength from the magnetron antenna. At the opposite end of the launcher section microwaves are transmitted in the transverse electric (TE) mode into the test chamber waveguide section where packed sorbent beds are placed in the microwave field. The launcher waveguide section is connected to a 60 dB directional coupler instrumented with a Hewlett-Packard (HP) 478A solid state microwave transducer and HP 432A power meter. Any microwave energy which has not been absorbed passes into a water load which serves as a sink for excess energy to prevent the reflection of microwaves backward through the waveguide components toward the magnetron. Water circulates through the load at a flow rate of $\approx$ 1 L/min under the action of a Micropump (Concorde, CA) #120-000 pump with #7144-00 gear drive. Microwave power output is controlled by a variable transformer which controls the voltage to the magnetron power supply. The relationship between variable transformer settings and output power is illustrated in Figure 2.

The packed sorbent beds are held in place within the test chamber using the mechanism illustrated schematically in Figure 3. The device is mounted vertically through the center of the test chamber waveguide section. The packed sorbent bed is confined within length of quartz tubing with an internal diameter of 1.07 cm, and held in place using glass wool end plugs. The sorbent bed is positioned so that the mid-point of the packed bed is in the exact center of

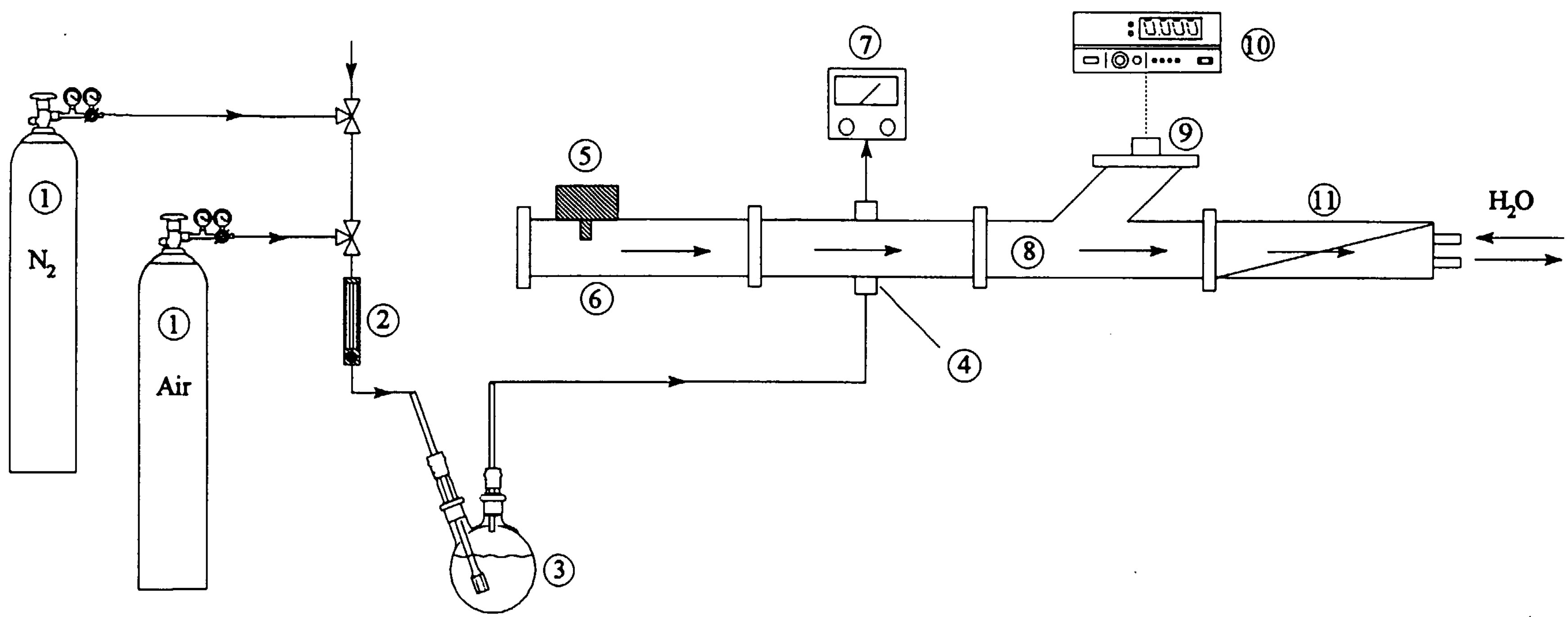


Figure 1. Microwave Powered Thermal Regeneration Experimental Apparatus. 1- Compressed Gas and Pressure Regulators, 2- Flow meter, 3-Sparger, 4-Sorbent Bed, 5-Magnetron, 6-Rectangular Waveguide, 7-Dew Point/Hydrocarbon/CO_2 Analyzer, 8-Directional Coupler, 9-Microwave Detector, 10- RF Power Meter, 11-Water Load.

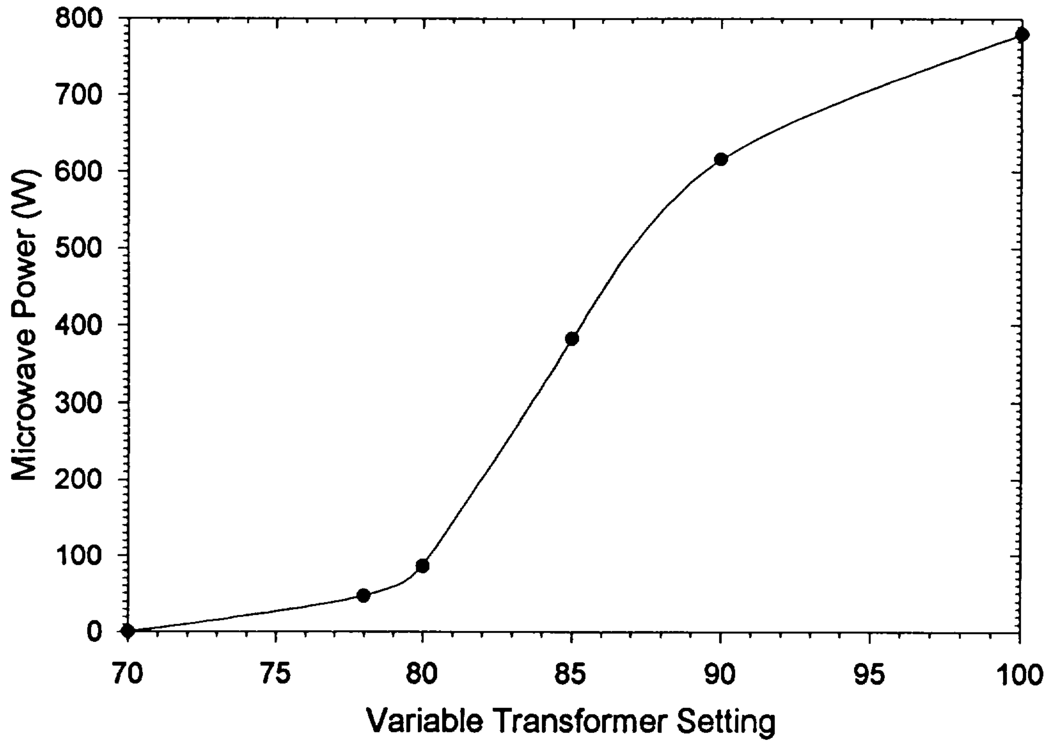


Figure 2. Microwave Power versus Variable Transformer Setting.

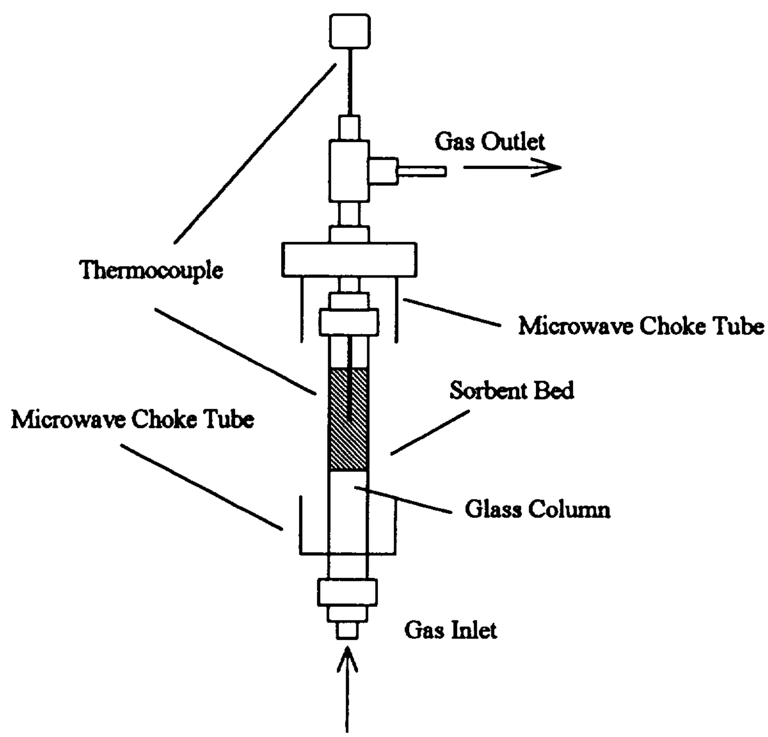


Figure 3. Device for Mounting Sorbent Beds in the Rectangular Waveguide.

the waveguide. Gas enters from the bottom and flows upward through the sorbent bed. A J-type thermocouple is mounted inside.

Challenge gases were fed into the apparatus from compressed gas cylinders, with initial pressures ranging from 15.5 - 20.7 MPa (2250 - 3000 psi). The gas stream passed through primary and second stage pressure regulators, to an Aalburg Instruments #052-04gG variable area flow meter (Cole-Parmer, Chicago, IL), and then through the sparging chamber illustrated in Figure 4, in which the stream was saturated with water vapor. In the case of CO_2 sorptions, the liquid within the sparging vessel was maintained at an acidic pH and was pre-saturated with the contaminant before flow into the packed sorbent bed was initiated. In the case of the organic contaminants, the aqueous phase consisted of a solution with an amount of dissolved organic compound sufficient for equilibrium to exist between the gas phase and the liquid phase, according to Henry's Law,

$$k_H = \frac{pC}{\chi}$$

where k_H is the Henry's Law constant, pC is the atmospheric partial pressure of the contaminant in atmospheres, and χ is the mole fraction of the dissolved contaminant.

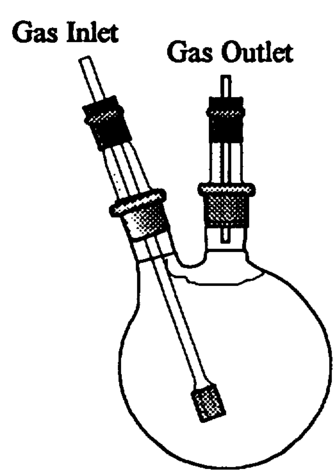


Figure 4. Sparging Apparatus for Humidifying the Contaminated Gas Stream.

Multifrequency Microwave Transmission and Reflection Apparatus. A network analyzer based system was assembled to measure the dielectric loss characteristics of candidate sorbents over the frequency range between 1.3 - 2.7 GHz. The apparatus is illustrated schematically in Figure 5. Variable frequency microwaves are output via coaxial cable from the Hewlett-Packard (HP) Model 8754A Vector Network Analyzer sequentially to: 1) a frequency doubler (MA-COM D-6-4), 2) an HP Model 778D dual directional coupler, and 3) a coaxial cable to WR 430 rectangular waveguide adapter. Attenuated signals representing both incident and reflected power levels are routed back to the network analyzer from the directional coupler. Microwaves are transmitted through the WR 430 rectangular waveguide in the transverse electric (TE) mode. The microwaves pass through a WR 430 to WR 650 waveguide transition and into the Specimen Confinement Chamber. The Specimen Chamber consists of a hollow rectangular box (18 x 12.9 x 3 cm) with an internal volume of 414 cm^3, constructed from 0.48 cm thickness polycarbonate sheets. The path length for microwave travel through the chamber is 2.05 cm. Microwaves reflected by the specimen travel in the reverse direction via the directional coupler to the network analyzer. Microwaves transmitted through the Specimen Confinement Chamber pass through a WR 650 to WR 430 transition into a WR 430 to coaxial cable adapter, and then by coaxial cable to the network analyzer. The network analyzer sweeps through the entire frequency range at a pre-determined sweep rate. Output power, reflected power, and transmitted power levels are monitored by the network analyzer and analog signals are output to a Hewlett-Packard Model 75900 X-Y plotter and also, via analog to digital converter (DATAQ DI180), to an IBM Compatible 80486 personal computer for data storage. In essence the waveguide sections form two symmetrical 'horn antennas' which surround the specimen and present the microwaves as a plane wavefront to the specimen. Dielectric loss characteristics are determined from a comparison of transmitted and reflected power, and from the related phase relationships, in comparison to the incident power.

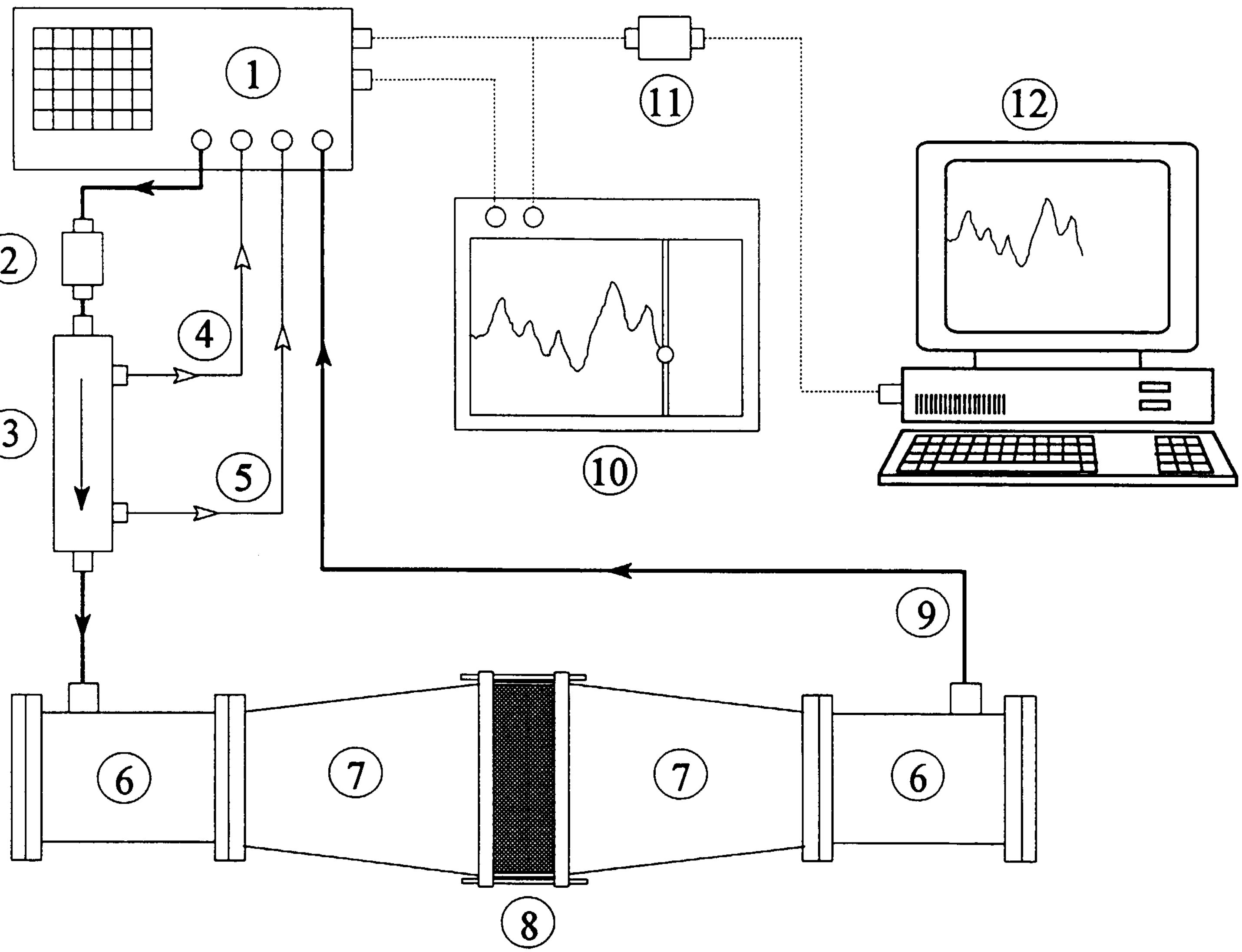


Figure 5. Multifrequency Microwave Transmission Test Apparatus: 1) Network Analyzer, 2) Frequency Doubler, 3) Dual Coaxial Directional Coupler, 4) Reference Power Signal, 5) Reflected Power Signal, 6) Coaxial Cable to WR 430 Waveguide Adapter, 7) WR 430 - WR 650 Waveguide Transition, 8) Specimen Housing, 9) Transmitted Power Signal, 10) X-Y Plotter, 11) Analog to Digital Converter, 12) Personal Computer.

Sorbents. Type 830IMC activated carbon was acquired from CECA, Inc. (Tulsa, OK). Coconut Shell Charcoal, #5769, was purchased from Alltech (Deerfield, IL). Carbosieve S-III carbon based molecular sieve was acquired from Supelco (Belefonte, PA). A second carbon based molecular sieve was prepared by Dr. Pramad Sharma at the Jet Propulsion Laboratory using the methods of Foley[10]. Lithium hydroxide was purchased from Cyprus Foote Mineral Company (Kings Mountain, NC). Molecular sieves 5A and 13X were donated by UOP (Des Plaines, IL). Silica gel was acquired from EM Science (Gibbstown, NJ). Silver carbonate was purchased from Aldrich (Milwaukee, WI). Zeolite ZSM-5 catalyst was donated by Mobil Corporation (Paulsboro, NJ). Pelletized silver oxide was prepared using a sodium silicate binder.

Contaminants. Compressed gas cylinders containing 0.5% carbon dioxide in air were obtained from Pacific Airgas (Portland, OR). Acetone and trichloroethylene were obtained from Aldrich (Milwaukee, WI). Separate contaminated air feed streams containing 300 ppm acetone and 50 ppm trichloroethylene (TCE) were prepared by addition of the appropriate volume of contaminant into evacuated high pressure gas cylinders, followed by pressurization with breathing quality air to 15.5 MPa (2250 psi) for steel cylinders and to 20.7 MPa (3000 psi) for aluminum cylinders.

Analytical Instruments. Carbon dioxide concentrations were determined using an ASTRO Model 2001 TIC/TOC analyzer (League City, TX) and an ASTRO 5600AT continuous on-line non-dispersive infrared (NDIR) CO_2 monitor. Trichloroethylene and acetone were determined using an HP-5710A gas chromatograph with a packed SP-1000 column (Supelco, Belefonte, PA) and flame ionization detection. Water vapor was monitored using an EG&G Model 880 Dew Point Hygrometer. Continuous hydrocarbon monitoring was performed using a Beckman Model 400 Hydrocarbon Analyzer.

III. RESULTS AND DISCUSSION.

Microwave Powered Thermal Regeneration of Sorbents.

The sorption and microwave powered thermal desorption characteristics of carbon dioxide, water vapor, acetone and trichloroethylene (TCE) were studied using a range of typical sorbent materials. Because of the diversity of the chemical properties of the contaminants, no individual sorbent was appropriate for the removal of all airborne species. The single-sorbent/single-contaminant experiments conducted are summarized in the matrix presented in Table I.

Table I - Summary of Individual Sorption/Regeneration Experiments.

	Acetone	CO_2	TCE	Water
Activated Carbon	+	-	+	-
Carbosieve S-III	+	-	+	-
Lithium Hydroxide	-	+	-	-
Molecular Sieve 5A	-	+	-	+
Molecular Sieve 13X	-	+	-	+
Silica Gel	-	-	-	+
Silver Oxide	-	+	-	-
Zeolite ZSM-5	+	-	+	-

Initial regeneration experiments using the waveguide based microwave irradiation apparatus were terminated prematurely due to the destruction of the thermocouple. Apparently, the grounded thermocouple positioned within the low loss material became an efficient absorber (antenna) and heated rapidly. This was confirmed when temperatures >1000 °C were indicated within a few seconds of full power irradiation of a LiOH bed confined within a quartz tube. In this case, it was evident that the temperature was that of the thermocouple, and not that of the packed bed. For this reason, in subsequent experiments the thermocouple was positioned in the exit gas stream. In this location, a few centimeters away from the sorbent bed and the microwave field, the temperature of the effluent gas emanating from the packed bed during regeneration was indicated. The experiments utilizing activated carbon were the single exception. Activated carbon was found to couple so strongly with the microwave radiation, that an embedded thermocouple was shielded and thus not affected.

Sorption and Thermal Desorption of Acetone. Activated carbon, Carbosieve S-III, and ZSM-5 were investigated as candidate thermally regenerable acetone sorbents for use with microwave powered heating systems. Based upon previous work, activated carbon and Carbosieve were known to sorb acetone strongly[11,12]. Further, Carbosieve S-III had been identified as a selective sorbent for low molecular weight species such as acetone and methyl-ethyl-ketone. In addition, the performance of ZSM-5, a high silica synthetic zeolite was also studied. ZSM-5 is commonly used as an industrial catalyst for a variety of synthetic reactions.

A 4.95 cm^3 packed bed of activated carbon (2.13 g) was prepared using #5769 coconut shell charcoal. In the initial sorption experiment, 300 ppm of acetone in air saturated with water vapor was fed to the sorbent bed at a flow rate of 1 L/min. Effluent acetone levels were monitored by an on-line hydrocarbon analyzer and confirmed by gas chromatography. Initial breakthrough was noted after 99 liters of flow. Fifty percent breakthrough occurred at 120 L. Complete breakthrough was observed after 150 L of cumulative flow. The loaded activated carbon bed was thermally regenerated using an initial power level of 23 W under a dry nitrogen flow at 0.1 L/min. At this relatively low level of incident radiation, bed temperatures of 180°C were attained. Upon increasing the power to 35 W, maximum bed temperatures of 380°C were observed. The adequacy of thermal regeneration was confirmed by a repeat sorption. Equivalent performance was attained prior to and after thermal regeneration. The results of sorption and regeneration experiments are presented in Figures 6 and 7, respectively.

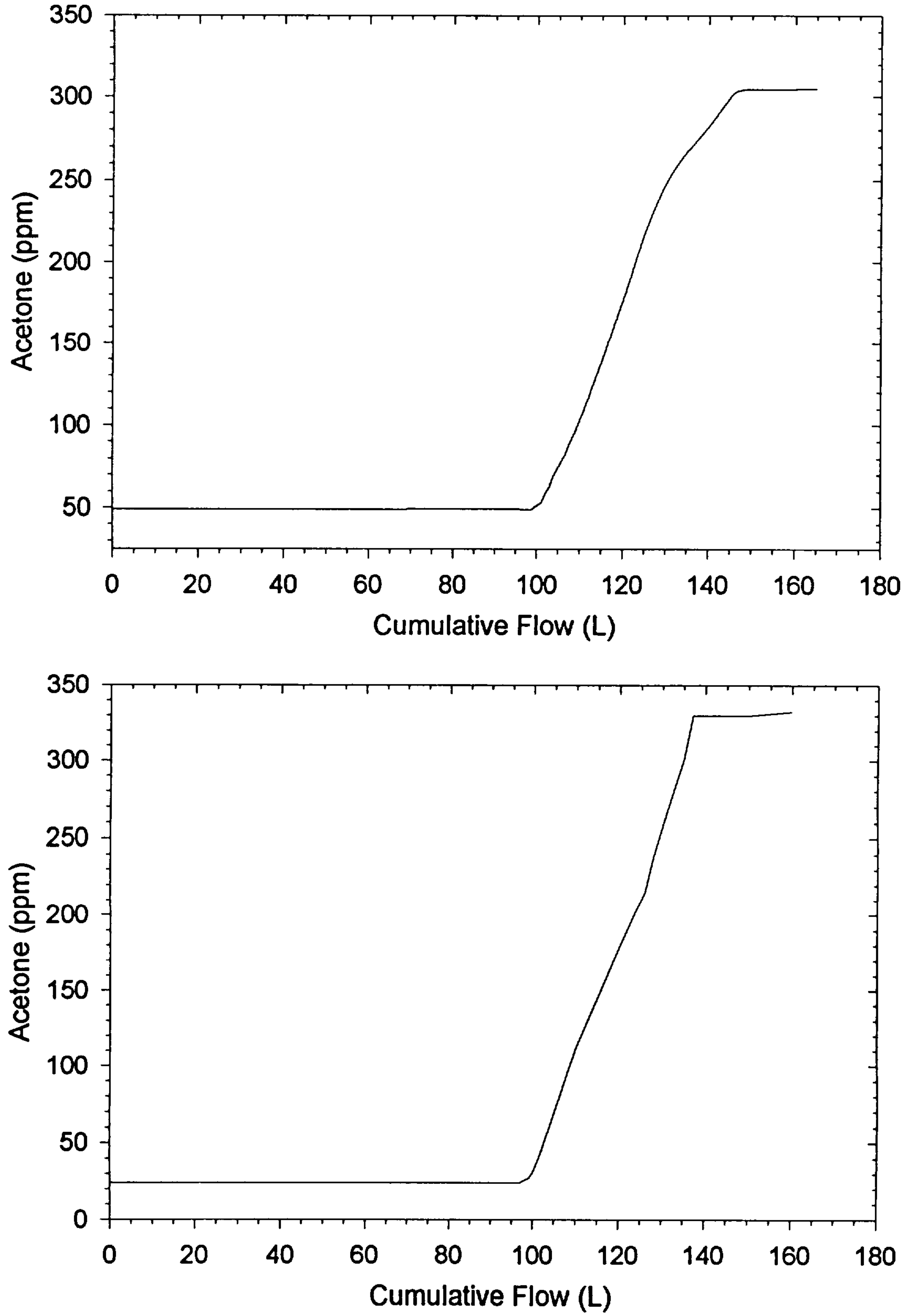


Figure 6. Sorption of Acetone on Activated Carbon: Top Initial, Bottom After Regeneration.

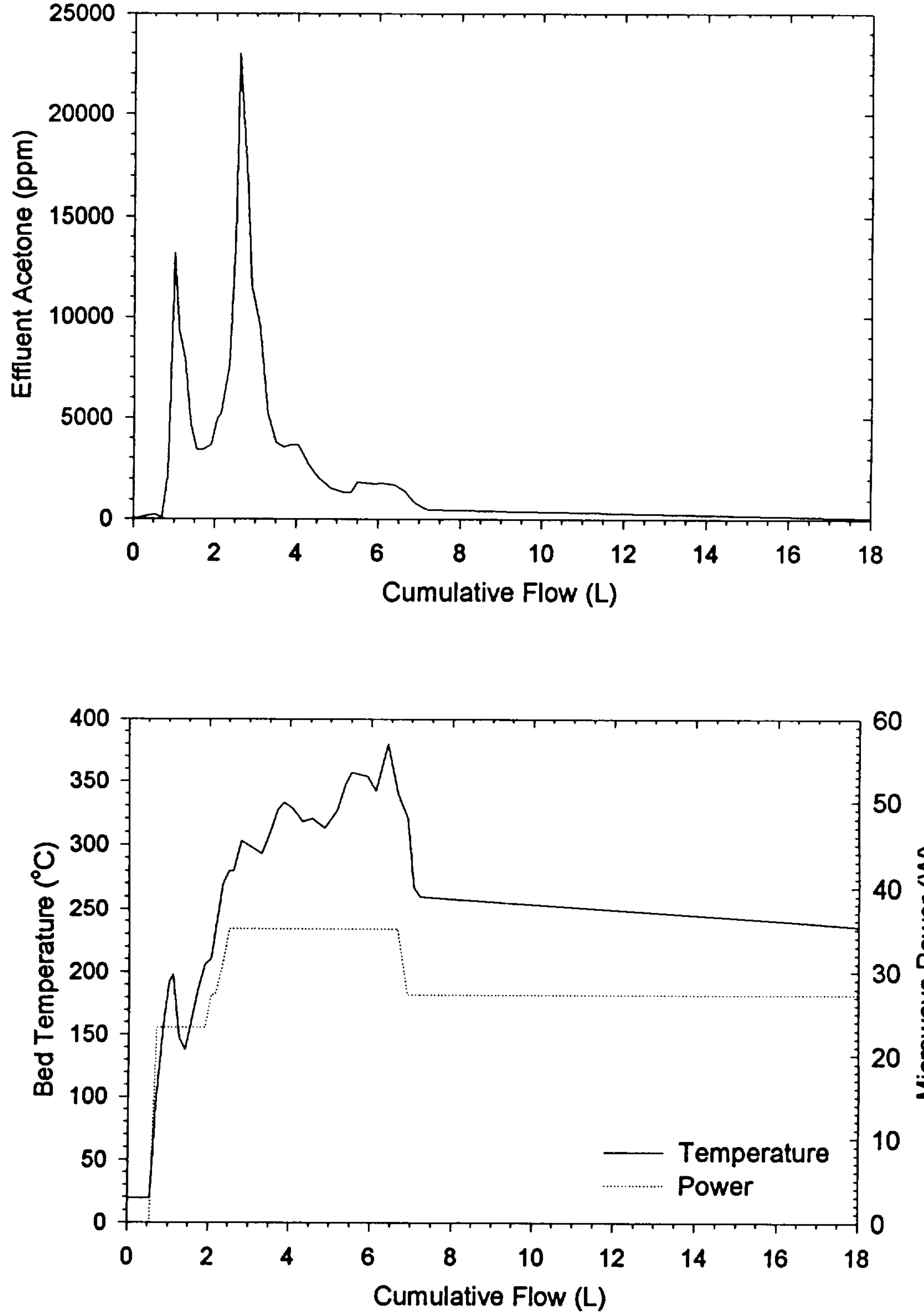


Figure 7. Thermal Regeneration of Acetone Loaded Activated Carbon.

A 1.9 cm^3 bed containing 1.36 g of Carbosieve S-III was challenged with humidified air containing ≈ 300 ppm acetone at 1 L/min. Initial breakthrough occurred after 90 liters of cumulative flow. Fifty percent breakthrough and total breakthrough were observed at 150 liters and 240 liters, respectively. The acetone loaded bed was thermally regenerated using stepwise increasing applied microwave power levels between 86 - 600 W, corresponding to exit gas temperatures between 90 - 230°C. The bulk of the acetone was desorbed at the 86 W power level. A subsequent sorption experiment confirmed the adequacy of the microwave powered thermal regeneration. Sorption and desorption experimental results are shown in Figures 8 and 9, respectively.

A 2.87 g packed bed containing 4.95 cm^3 of ZSM-5 catalyst in the hydrogen form was challenged with ≈ 240 ppm acetone in humidified air at 1 L/min. Breakthrough began after 20 L of flow. Fifty percent breakthrough was observed at 83 L, and total breakthrough occurred after 160 L of cumulative flow. The bed was thermally regenerated at full microwave power (780 W). The thermocouple was located in the exit gas stream, and indicated a maximum temperature of ≈ 80°C. A repeat sorption experiment was conducted using 236 ppm acetone in humidified air. The breakthrough characteristics observed during the second run indicated that the ZSM-5 bed had been adequately regenerated. The results of the sorption and regeneration experiments are presented in Figures 10 and 11, respectively.

Sorption and Thermal Desorption of Carbon Dioxide. CO_2 sorption experiments were conducted using lithium hydroxide, Molecular Sieve 5A, the carbogenic molecular sieve prepared at JPL, silver carbonate, and silver oxide.

A lithium hydroxide bed containing 1.62 g was challenged with 0.5% CO_2 in dry air at 1 L/min. Effluent CO_2 levels were determined on individual samples using a TIC/TOC analyzer. A thermal regeneration was attempted for 15 minutes at 780 W microwave power using dry nitrogen. In this experiment maximum effluent gas temperatures reached 51°C after eight minutes and remained steady thereafter. The total effluent was collected

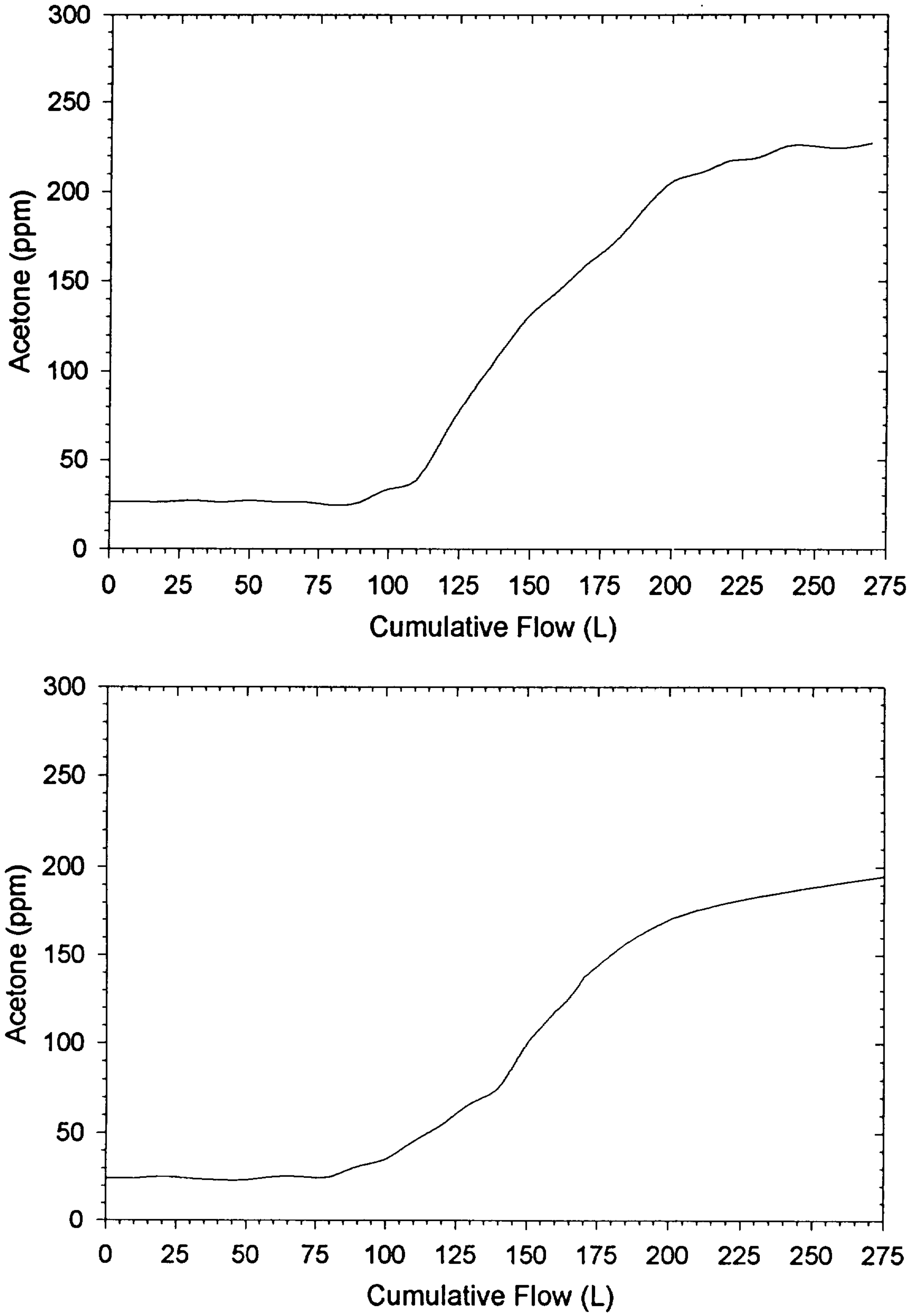
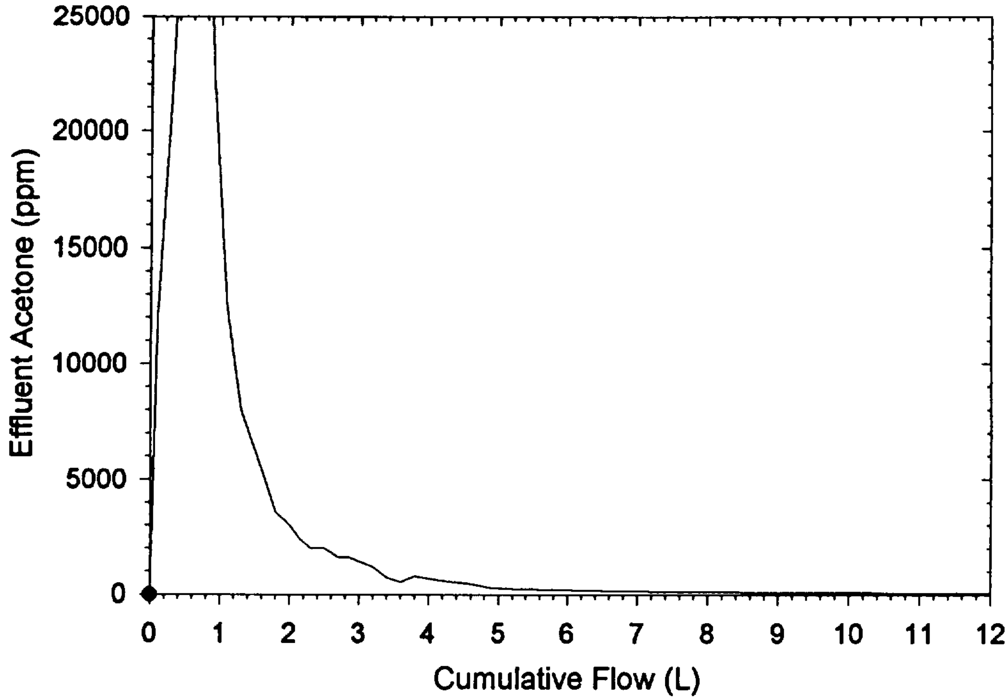


Figure 8. Sorption of Acetone on Carbosieve S-III: Top Initial, Bottom After Regeneration.



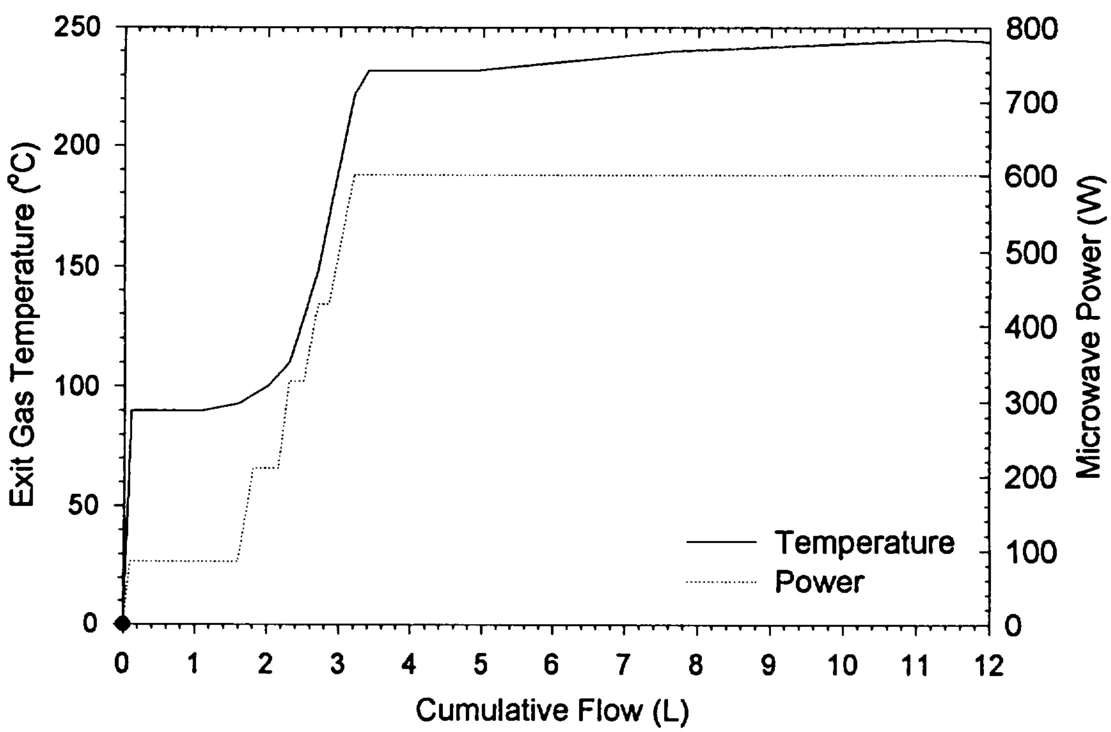


Figure 9. Thermal Regeneration of Acetone Loaded Carbosieve S-III.

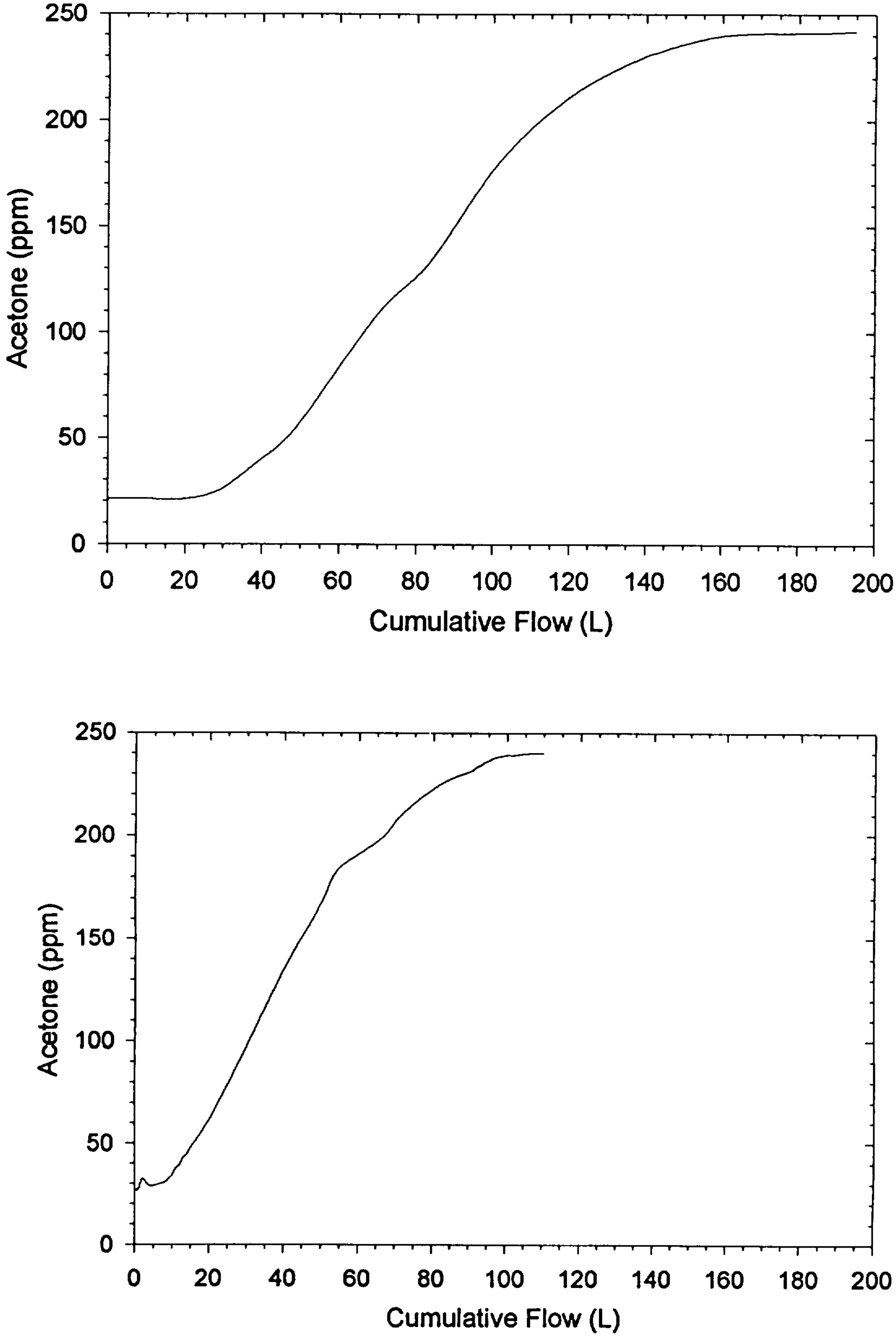


Figure 10. Sorption of Acetone on Zeolite ZSM-5: Top Initial, Bottom After Regeneration.

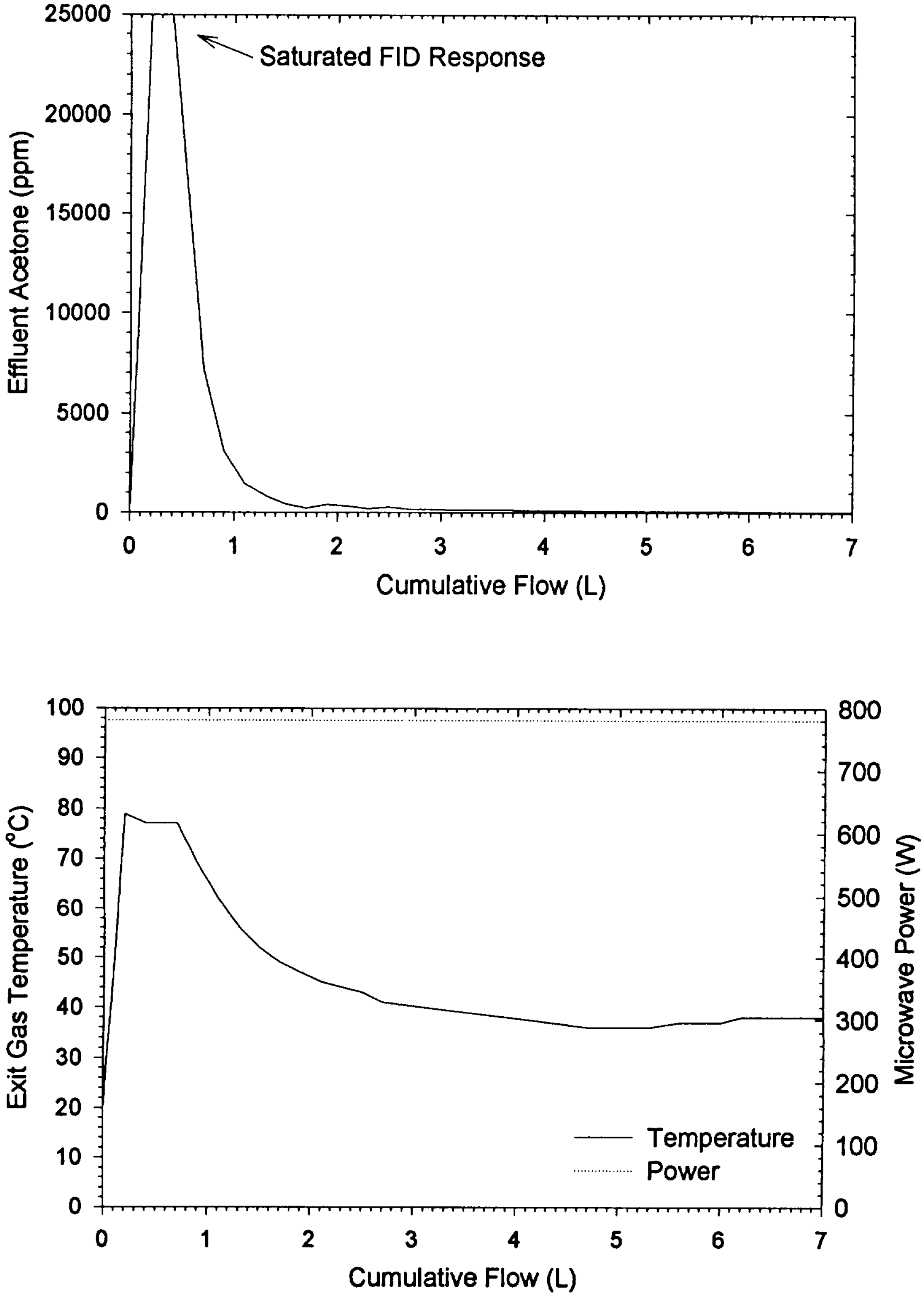


Figure 11. Thermal Regeneration of Acetone Loaded ZSM-5.

in a gas-tight bag which yielded a CO_2 concentration of 0.04%. Following this regeneration attempt, the bed was challenged with 0.5% CO_2 in air saturated with water vapor. Under these conditions additional CO_2 was adsorbed. This is believed to be due to the facilitation of the chemisorption reaction by the presence of water. For this reason, the sorption from humidified air was repeated using a fresh LiOH bed. The results of the dry and wet air sorptions are presented in Figure 12. This was followed by a second attempted thermal desorption using nitrogen saturated with water vapor. The effluent gas reached a maximum temperature of 55° C. The effluent gas yielded an average CO_2 concentration of 0.06%. While the LiOH bed coupled with the microwave field to a minor extent, temperatures achieved were not sufficient to reverse the chemisorption of CO_2.

In order to achieve the higher temperatures required for reversal of the reactions responsible for chemisorption of CO_2 by LiOH, a composite bed was prepared consisting of 90% LiOH and 10% activated carbon by weight. This bed was exposed to the full microwave power of the waveguide irradiation system (780 W). Under these conditions, the quartz tube melted after an exposure of less than one minute, indicating that bed temperatures in excess of 1470°C had been attained. Clearly, very high temperatures can be achieved using a composite bed. However, owing to the constraints of time, this line of investigation was discontinued.

A packed bed containing 2.81 g of Molecular Sieve 5A was challenged with dry air containing 0.5% CO_2 at a flow rate of 1 L/min. The initial breakthrough occurred almost immediately. Fifty percent breakthrough occurred at approximately 6 liters of cumulative flow. Total breakthrough was observed after 50 liters. Regeneration was conducted using full microwave power and dry nitrogen at 0.1 L/min. Gases were collected in impermeable bags for analysis. During thermal desorption the effluent gas temperature rose to a maximum of 55° C. The results of sorption and regeneration experiments are shown in Figure 13. Integration of the breakthrough curve yielded a total mass loading of 56 mg CO_2, corresponding to 2% of the initial sorbent bed weight. The microwave powered thermal desorption yielded 75 mg CO_2. The discrepancy is believed due to the sorption of CO_2 by the sorbent bed during

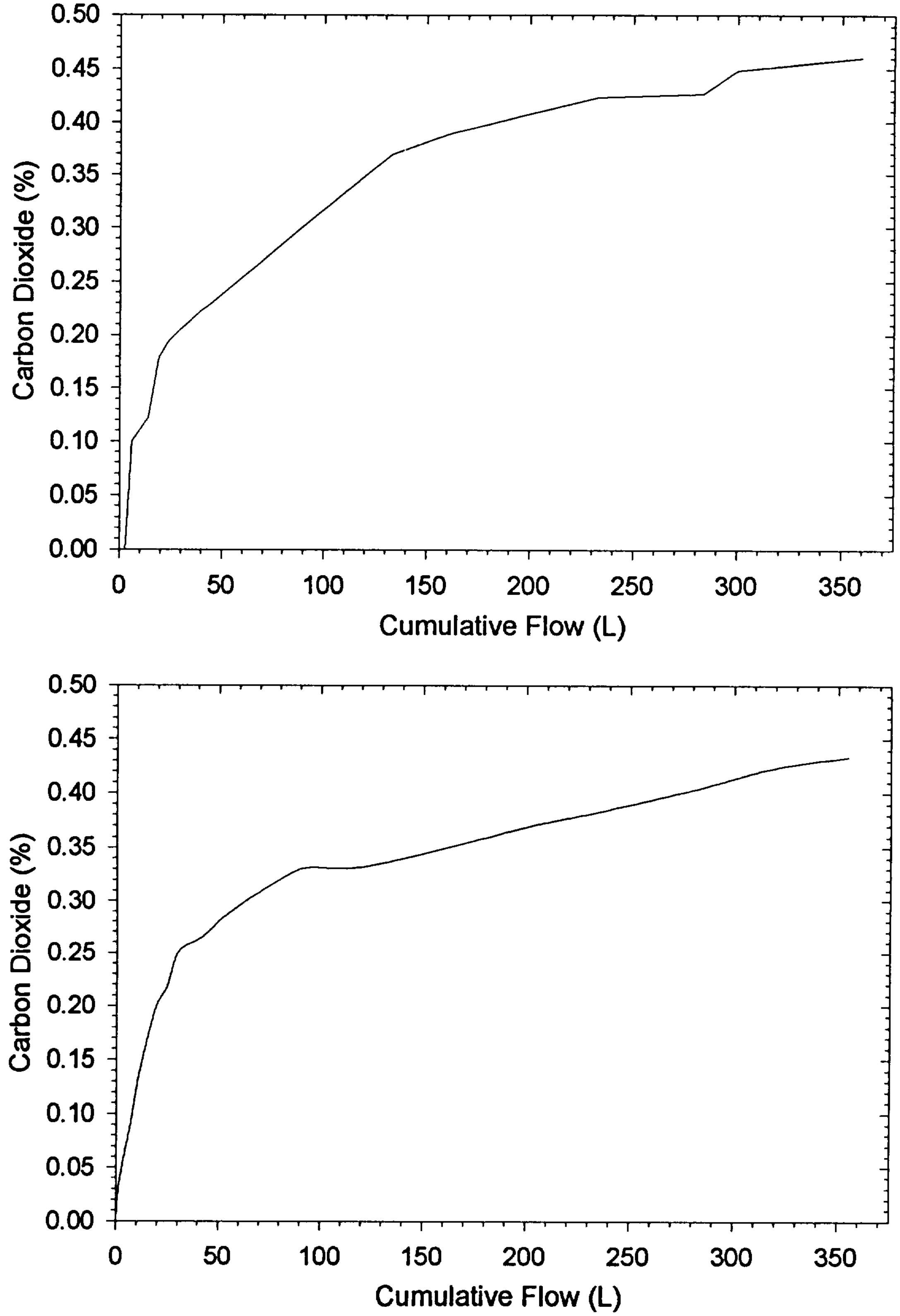


Figure 12. Chemisorption of CO_2 by LiOH: Top-dry sorption, Bottom-wet sorption.

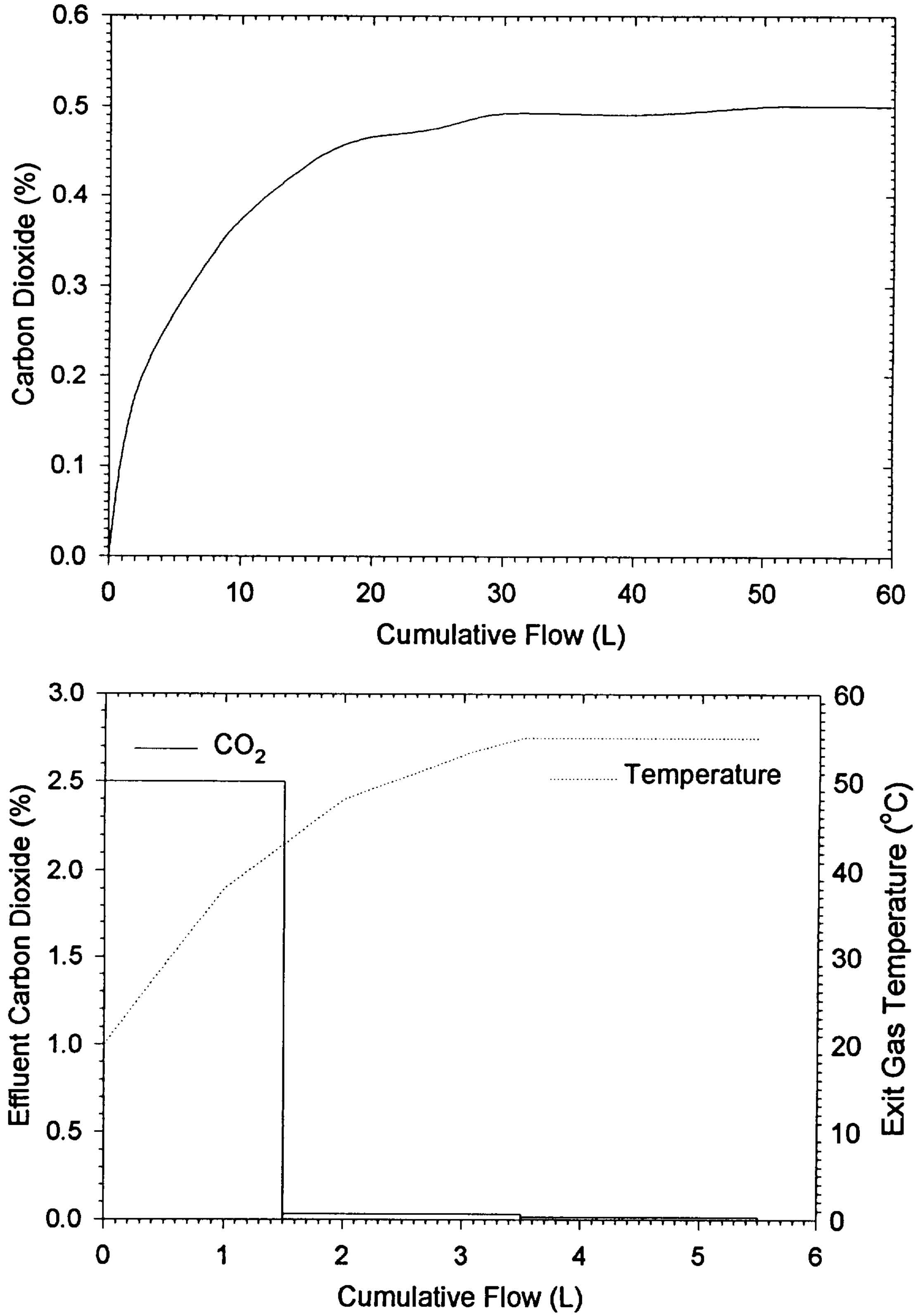


Figure 13. Sorption of CO_2 by Molecular Sieve 5A (top), and Regeneration (bottom).

exposure to the atmosphere prior to initiation of the experiment. The carbon dioxide levels recovered upon heating indicated a complete regeneration of the Molecular Sieve 5A.

A 1.8 cm^3 packed bed containing the total supply of carbogenic molecular sieve prepared for us by JPL (0.52 g) was challenged with humidified air containing 0.5% CO_2 at 1 L/min. The first sample of effluent was collected after 2 L of cumulative flow and indicated total breakthrough of CO_2. The subsequent thermal desorption experiment was invalidated because the glass wool plug at the outflow face of the bed melted, and the packed bed was pushed beyond the microwave radiation field inside the waveguide. It is noteworthy that the softening point for borosilicate glass is $\approx$ 700°C. This temperature was apparently achieved inside the packed bed, while the indicated exit gas temperature never exceeded 92°C. This result suggests strong coupling between the carbogenic molecular sieve and the incident 2.45 GHz radiation. The bed was repacked and the glass wool plugs were repositioned so that they did not make physical contact with the sorbent bed, and also were located outside the microwave field. Preliminary tests indicated that the repacked bed was neither moved nor fluidized by the 1 L/min air flow.

The sorption experiment was repeated with identical results; after 2 minutes (the time of the first sample collection) total breakthrough of CO_2 was evident. The subsequent regeneration was conducted under reduced power ($\approx$ 390 W) to avoid the extreme temperatures encountered previously. Thermal desorption was conducted under dry nitrogen flowing at 0.1 L/min. A maximum outlet gas temperature of 57°C was observed. The effluent gas was collected in 1 L gas-tight bags at 10 minute intervals. The analysis of the first sample indicated 0.13% CO_2. Subsequent samples contained no carbon dioxide. Assuming ideality and a temperature of 20°C inside the gas bag, this corresponds to 2.38 mg of CO_2 (0.5% of bed weight).

Following this, a third sorption was conducted. This time total breakthrough was not achieved until 7 minutes into the experiment. The subsequent thermal desorption yielded 5.49 mg CO_2, corresponding to approximately 1% of the sorbent bed's weight. Performance of the sorbent was apparently improving over subsequent regenerations. It is not known whether the performance of this material was adversely affected by the temperatures ≥ 700°C achieved during the first series of experiments. Also, considering the very small volume of sorbent available, the very high face velocity, and the highly unfavorable ratio of bed length to volume under which these tests were conducted, that any CO_2 sorption was observed must be taken as a preliminary positive result for this material. Further work with sufficient quantities of material of known pore size distribution should be conducted.

As an initial evaluation of the potential for microwave powered regeneration of silver oxide based CO_2 sorbents, a packed bed containing 0.68 g of powdered reagent grade Ag_2CO_3 was irradiated at full power (780 W) under a dry nitrogen flow of 0.1 L/min. No increase in the exit gas temperature above ambient was observed after 5 minutes. Following this experiment a bed containing 2.00 g of Ag_2O pellets prepared using a sodium silicate binder was irradiated for four minutes under similar conditions. The exit gas temperature stabilized at 43°C. The bed was quickly opened and a surface temperature of 159°C was indicated by an IR thermometer. Grain boundary effects are know to be important in dielectric loss mechanisms. This may explain the difference in behavior between the powder and the pellets.

While the heating achieved in the silver oxide pellets under microwave irradiation was significant, the temperatures achieved were still less than the typical 220°C at which silver oxide sorbents are regenerated. To explore the possibility of attaining higher temperatures, a 3.60 cm^3 composite bed containing 1.14 g of silver carbonate and 0.76 g of activated carbon was prepared. This bed was irradiated using a stepwise increase in microwave power under dry nitrogen flowing at 0.1 L/min. The effluent was collected in gas-tight bags and analyzed. Considerable carbon dioxide was produced. The experimental results are shown in Figure 14. Maximum exit gas temperatures of 183°C were recorded at full power. Inspection of the bed after irradiation indicated that the silver carbonate had been reduced to metallic silver. This was most probably because decomposition temperatures (300°C) were achieved within the bed. The reducing activity of the activated carbon may have been a contributing factor.

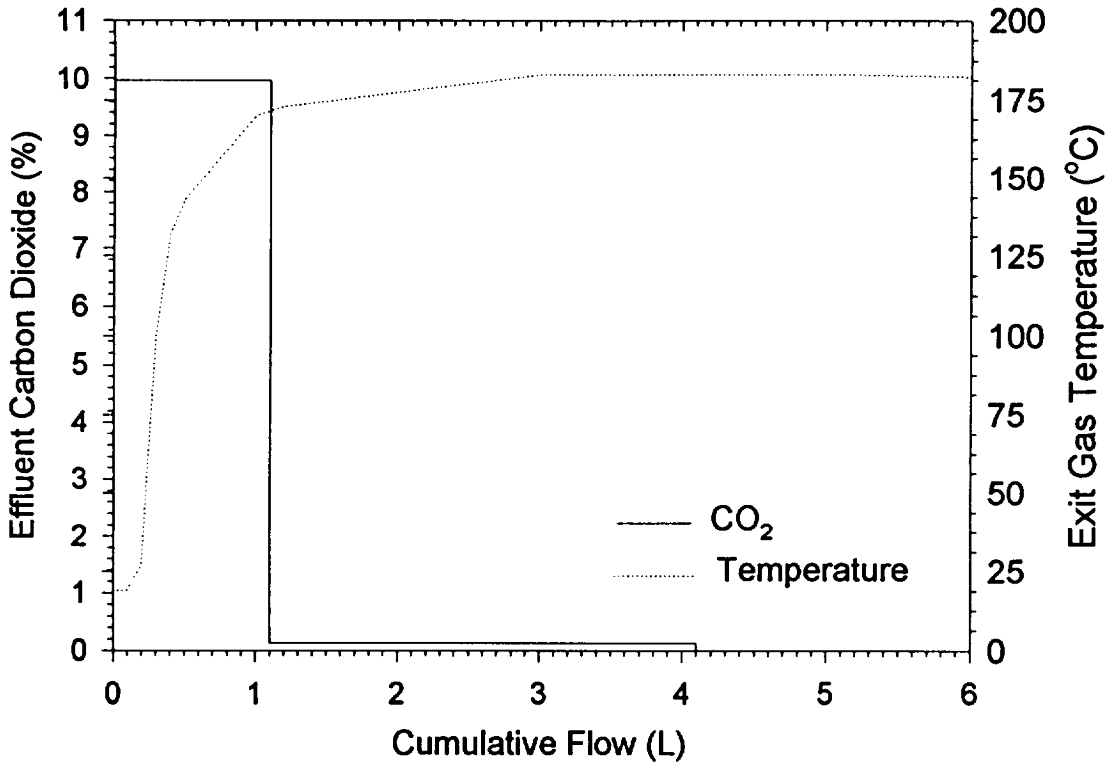


Figure 14. Microwave Powered Thermal Decomposition of Silver Carbonate.

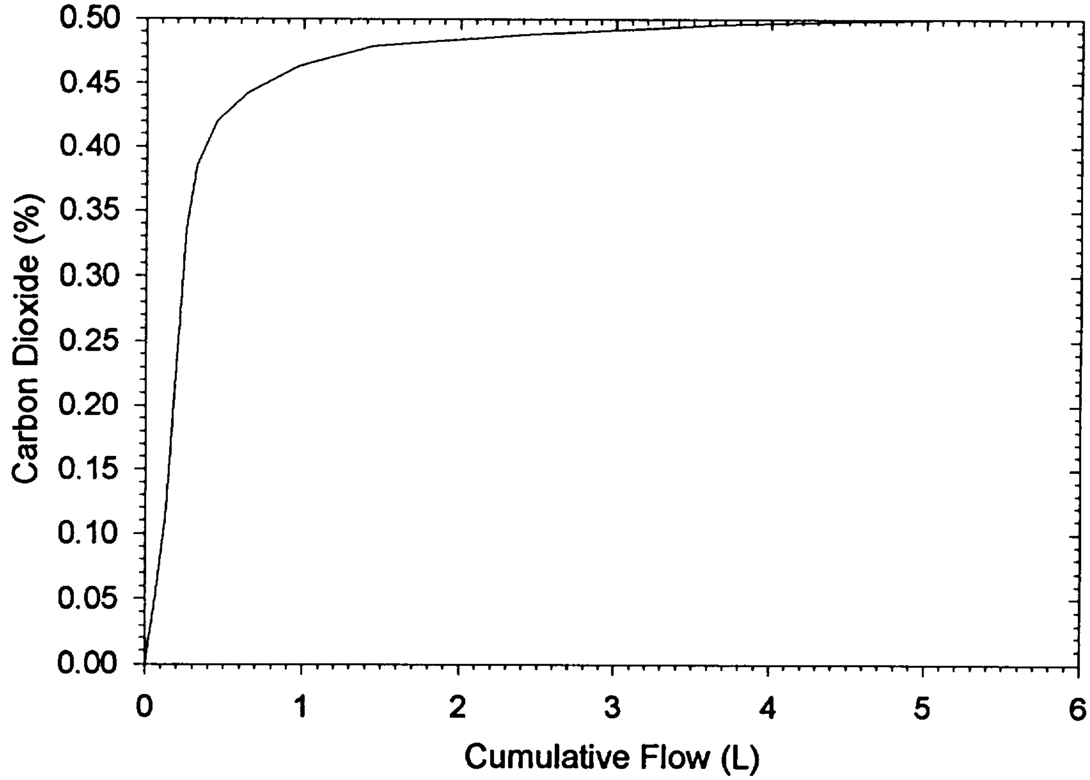
A 2.97 cm³ composite bed was prepared containing 2.00 g of Ag_2O pellets and 2.27 g of powdered α-silicon carbide. The initial brownish coloration of the Ag_2O indicated that the sorbent was at least partially loaded with CO_2. The Ag_2O-SiC composite bed was irradiated at 245 W with water saturated nitrogen flowing at 0.1 L/min and effluent CO_2 levels were monitored continuously. Immediately upon application of power, effluent CO_2 levels rose above the 5,000 ppm upper limit of the NDIR detector and remained off scale for sixteen minutes. An effluent carbon dioxide concentration of ≈ 24,500 ppm was determined for a gas sample collected during this period. Exit gas temperature reached a maximum of ≈ 72°C. Following regeneration, a bed temperature of 269°C was measured using an IR thermometer. This indicated that temperatures were probably not high enough within the bed to promote decomposition of the silver oxide during the thermal desorption. The regenerated Ag_2O-SiC bed was challenged with 0.5% CO_2 in humidified air at 0.32 L/min. Fifty percent breakthrough occurred in 1.5 minutes. Seventy five percent breakthrough was seen at 16 minutes, and complete breakthrough was evident at 50 minutes.

To investigate the compatibility of a molecular sieve CO_2 sorbent and a strong microwave susceptor, a composite bed containing 1.05 g Molecular Sieve 5A and 0.87 g activated carbon was challenged with 0.5% carbon dioxide in dry air at a flow rate of 320 mL/min. Effluent CO_2 levels were tracked continuously using an on-line NDIR analyzer. Following sorption, the bed was thermally regenerated using microwave power levels ranging from 39 - 210 W. The adequacy of thermal regeneration was confirmed by a second sorption, yielding virtually identical results. The sorption and regeneration experimental results are illustrated in Figures 15 and 16, respectively.

Sorption and Thermal Desorption of Trichloroethylene (TCE). The sorption and microwave powered thermal desorption characteristics of trichloroethylene (TCE) were studied using activated carbon, Carbosieve S-III, and ZSM-5.

A 4.95 cm^3 activated carbon bed weighing 2.21 g was challenged with ≈ 50 ppm TCE in humidified air at a flow rate of 1 L/min. In this experiment, sorption was limited by kinetics. Initial steady-state effluent TCE concentrations were ≈ 6 ppm and began rising to higher levels between 400 - 500 L of cumulative flow. The TCE loaded activated carbon bed was regenerated under nitrogen at 0.1 L/min. During regeneration a maximum sorbent bed temperature of 525°C was observed at a microwave power level of 38 W. Following thermal regeneration, a second sorption was conducted, yielding results similar to that of the first sorption, indicating that a complete reactivation of the bed had been achieved. The two sorption events are depicted in Figure 17. Concentration, temperature and power profiles for the microwave powered thermal desorption are illustrated in Figure 18.

An attempt was made to load a 5 cm^3 bed containing 3.46 g of Carbosieve S-III. Challenged with 50 ppm TCE, breakthrough had not begun after 1255 L of cumulative flow.



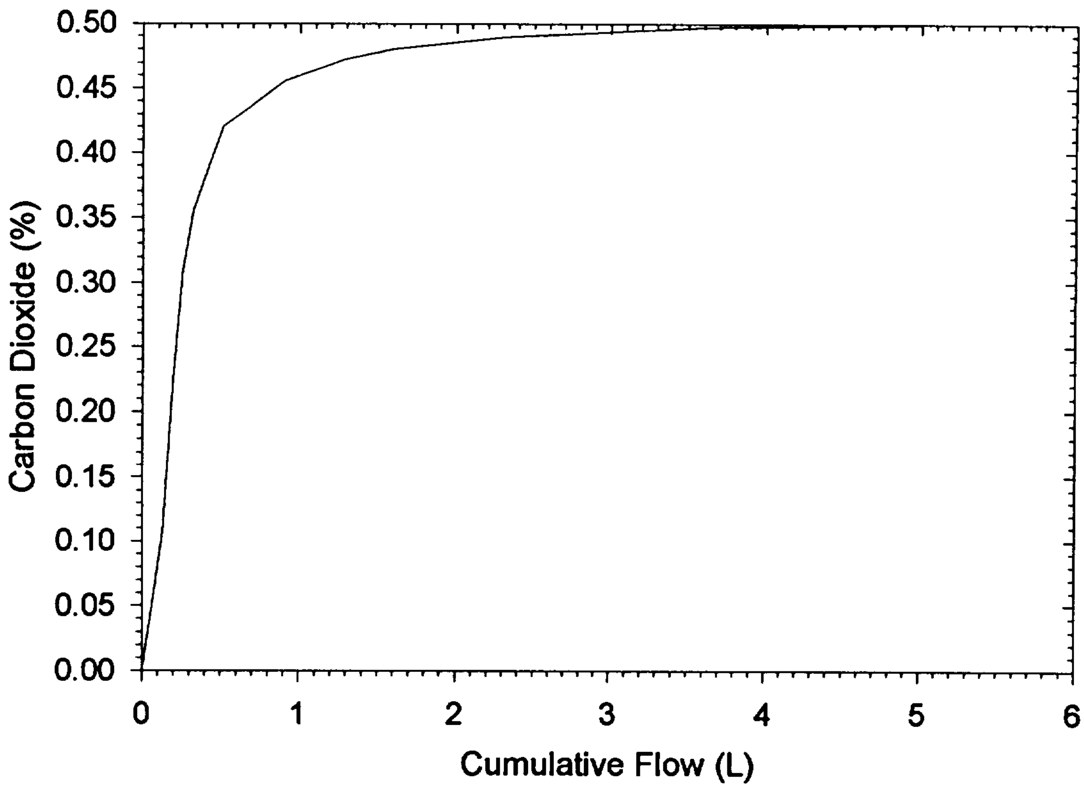


Figure 15. Sorption of CO_2 on Composite MS-5A: Top - Initial, Bottom -After Regeneration.

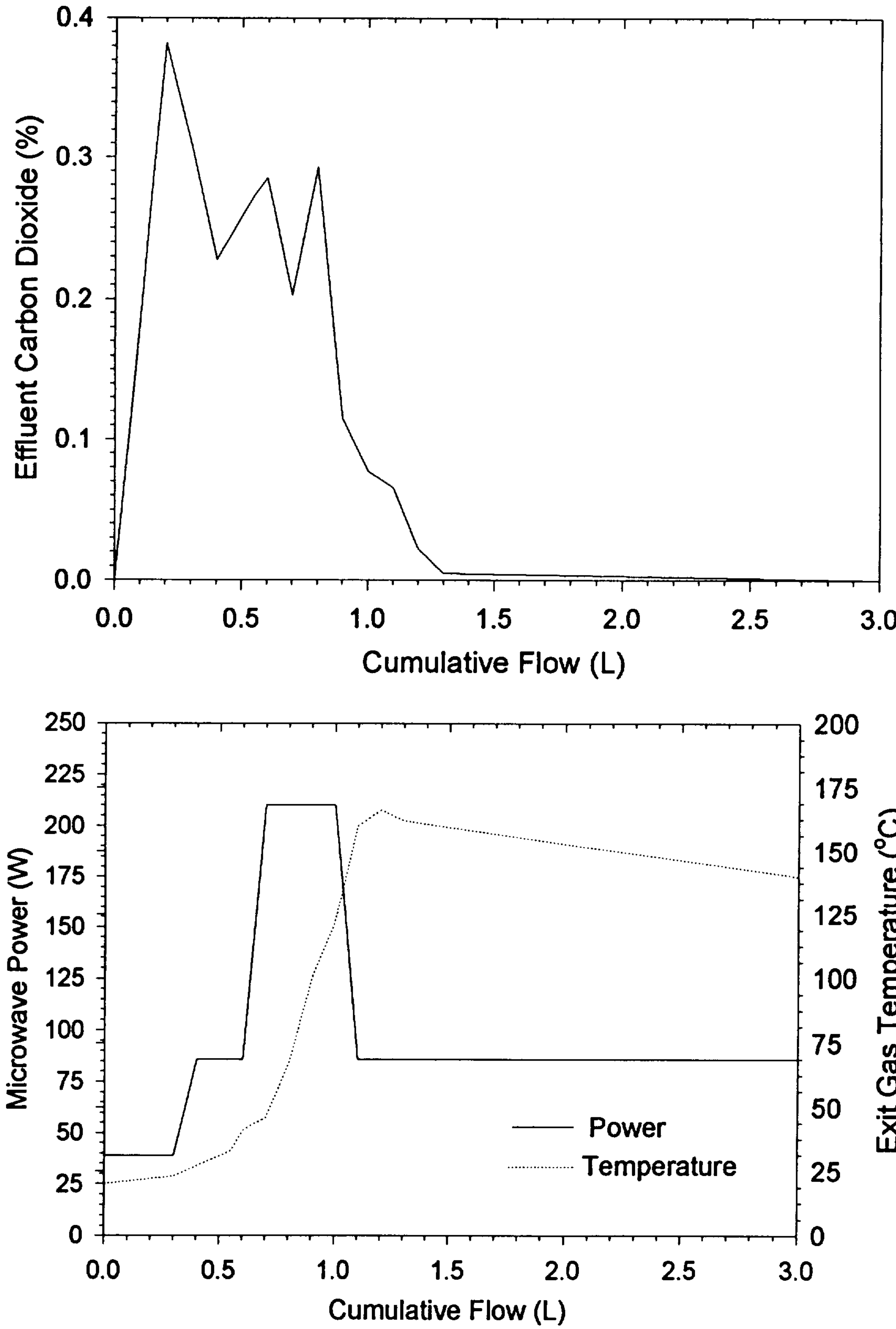


Figure 16. Thermal Regeneration of CO_2 Loaded MS 5A Composite Bed.

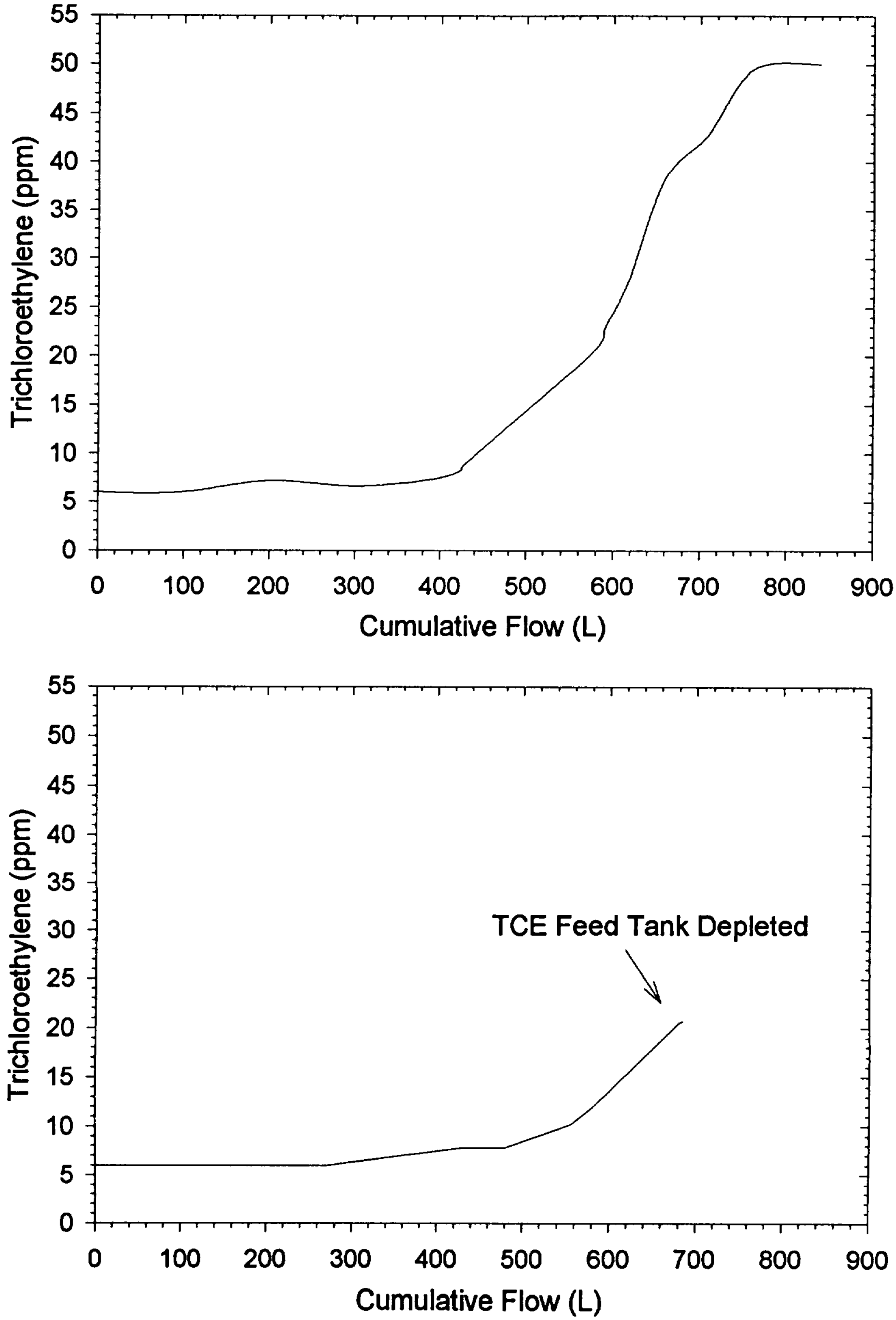


Figure 17. Sorption of TCE on Activated Carbon: Top Initial, Bottom After Regeneration.

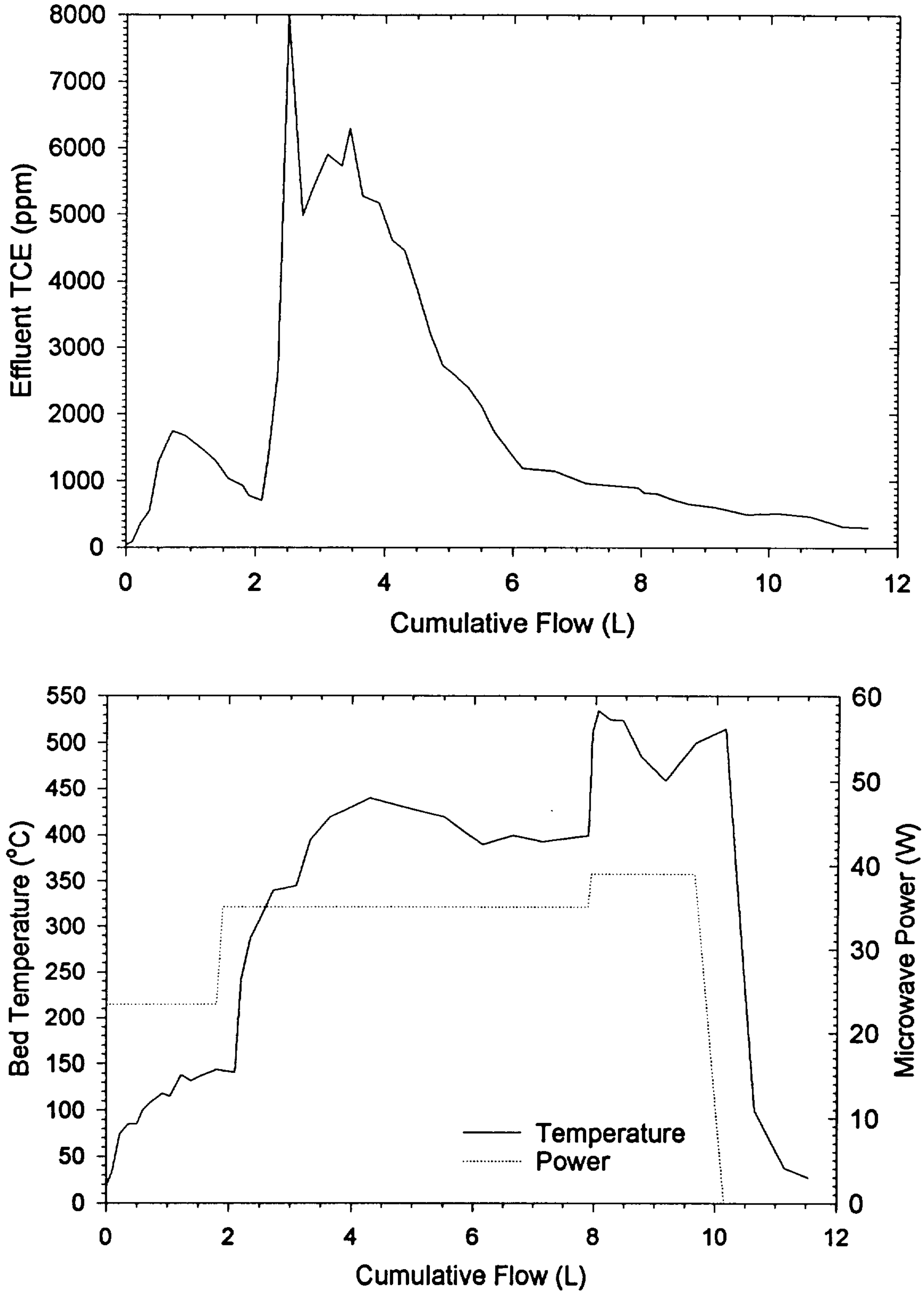


Figure 18. Regeneration of TCE Loaded Activated Carbon.

low. Due to the apparently extremely high capacity of this sorbent for TCE, a smaller bed containing only 0.33 g was prepared. Breakthrough for this bed began at approximately 30 L of cumulative flow. Thermal desorption was conducted at a constant 490 W power level, during which a maximum effluent gas temperature of 63°C was observed. The repeat sorption experiment (40 ppm TCE) confirmed that the bed had been adequately regenerated. The results of sorption and thermal regeneration experiments are presented in Figures 19 and 20.

A 2.77 g bed of hydrogen form ZSM-5 was challenged with 550 ppm TCE in humidified air at 1 L/min. Owing to a calculational error, the contaminated air for this experiment was prepared at a much higher concentration than intended. Complete breakthrough occurred immediately upon initiation of flow. The loaded bed was thermally desorbed under full microwave power using dry nitrogen at 0.1 L/min. The exhaust gas temperature rapidly heated to 90°C, and then slowly fell to 35°C. The initially high temperature can be attributed to the direct coupling of the incident microwaves with both adsorbed water and TCE. Virtually all TCE was desorbed in the first 800 cm^3 of effluent gas. The results of the thermal desorption are illustrated in Figure 21.

Sorption and Thermal Desorption of Water Vapor. Water loaded silica gel, Molecular Sieve 13X, and Molecular Sieve 5A were evaluated for compatibility with microwave powered thermal regeneration.

A packed bed containing ≈ 5 cm^3 of silica gel was installed in the waveguide. Breathing quality air flowed through the sparging apparatus and into the packed bed at a flow rate of 1 L/min. Effluent water vapor was tracked using an on-line dew point monitor. Measurements taken with the air flow by-passing the sorbent bed indicated that the gas stream was fully saturated with water vapor. The initial sorption of water vapor on silica gel is illustrated in Figure 22. The sorption was strongly exothermic, yielding a maximum exit air temperature of 42°C. Following sorption, air flow was terminated and a flow of dry nitrogen was initiated at

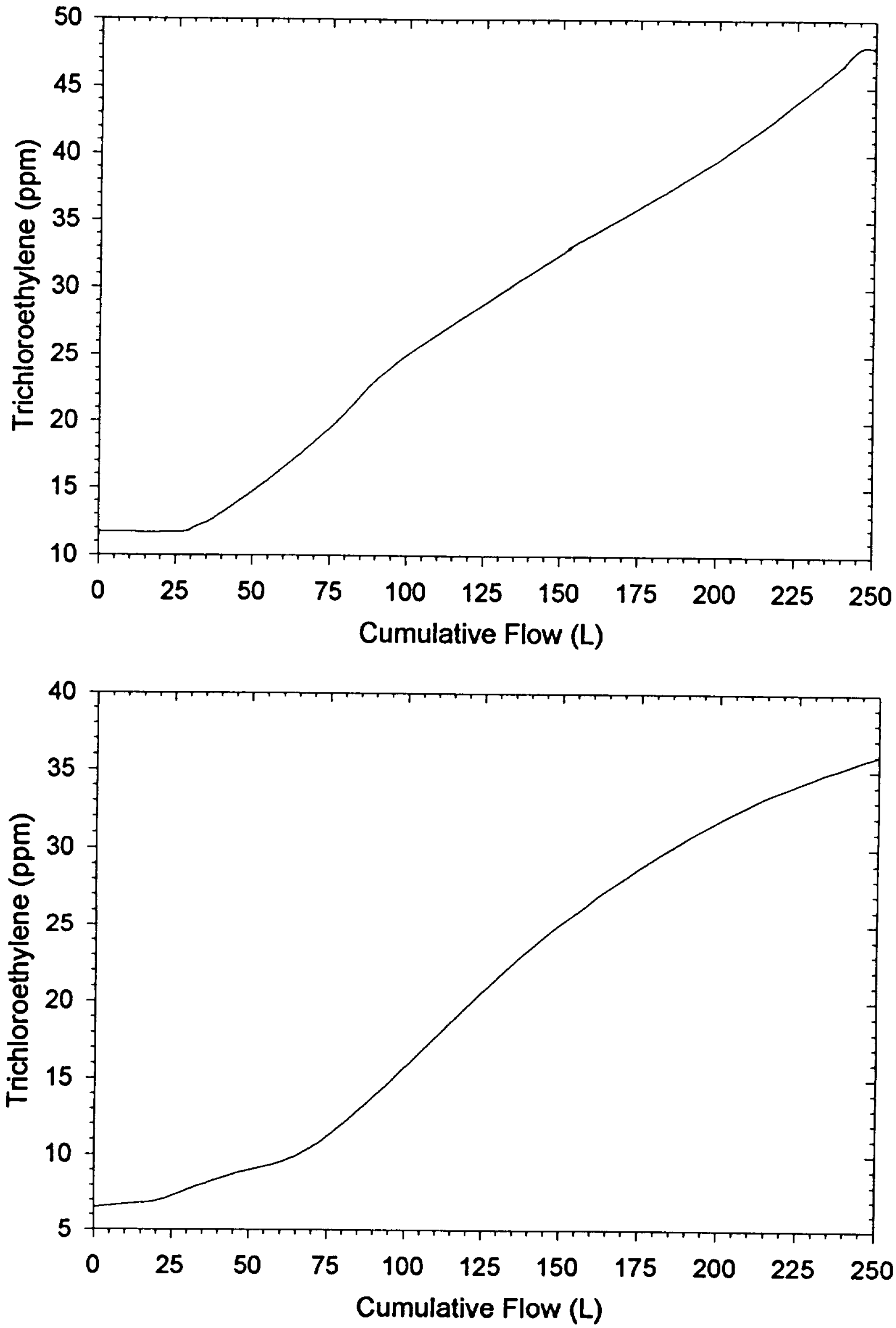


Figure 19. Sorption of TCE on Carbosieve S-III: Top Initial, Bottom After Regeneration.

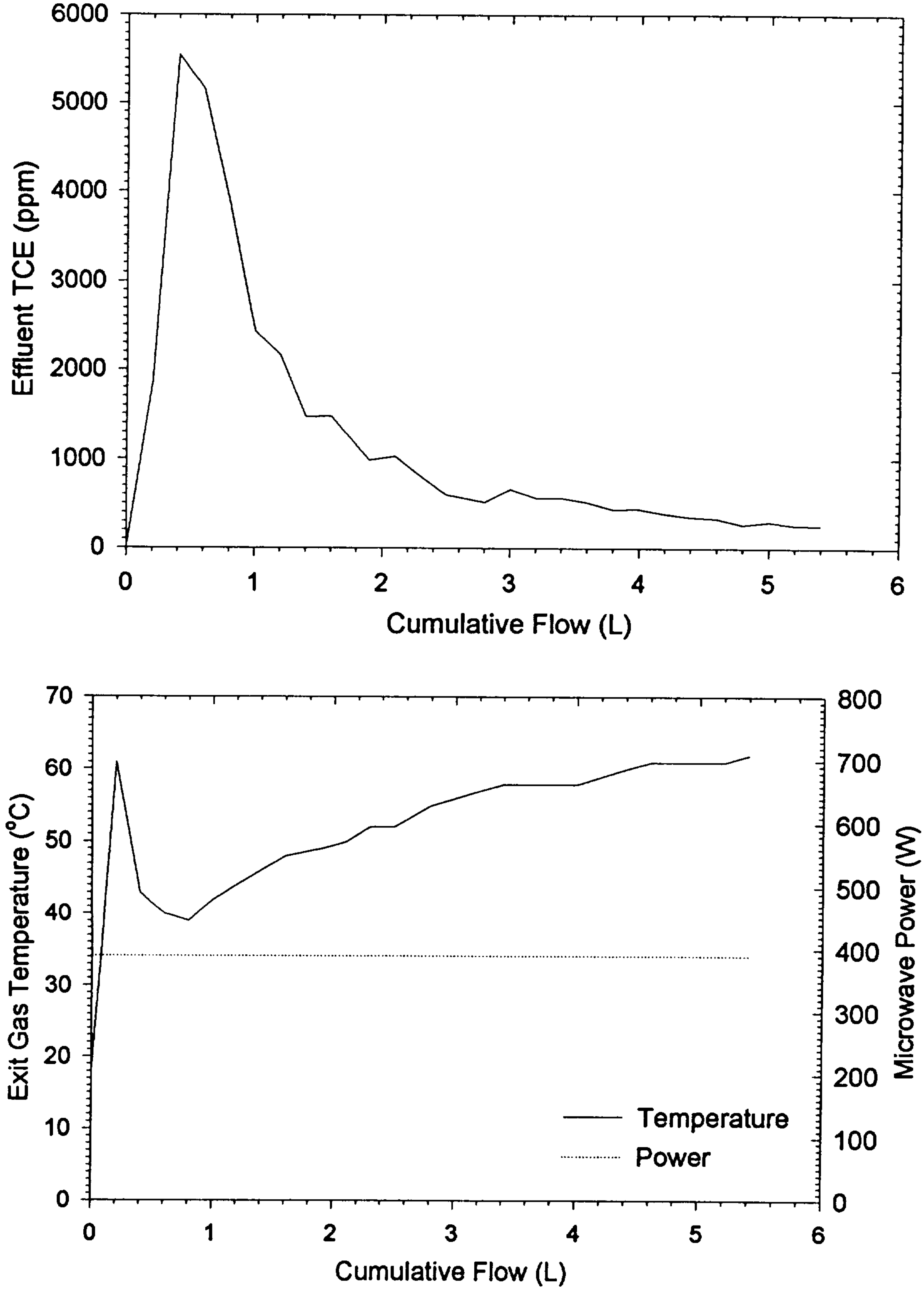


Figure 20. Regeneration of TCE Loaded Carbosieve S-III.

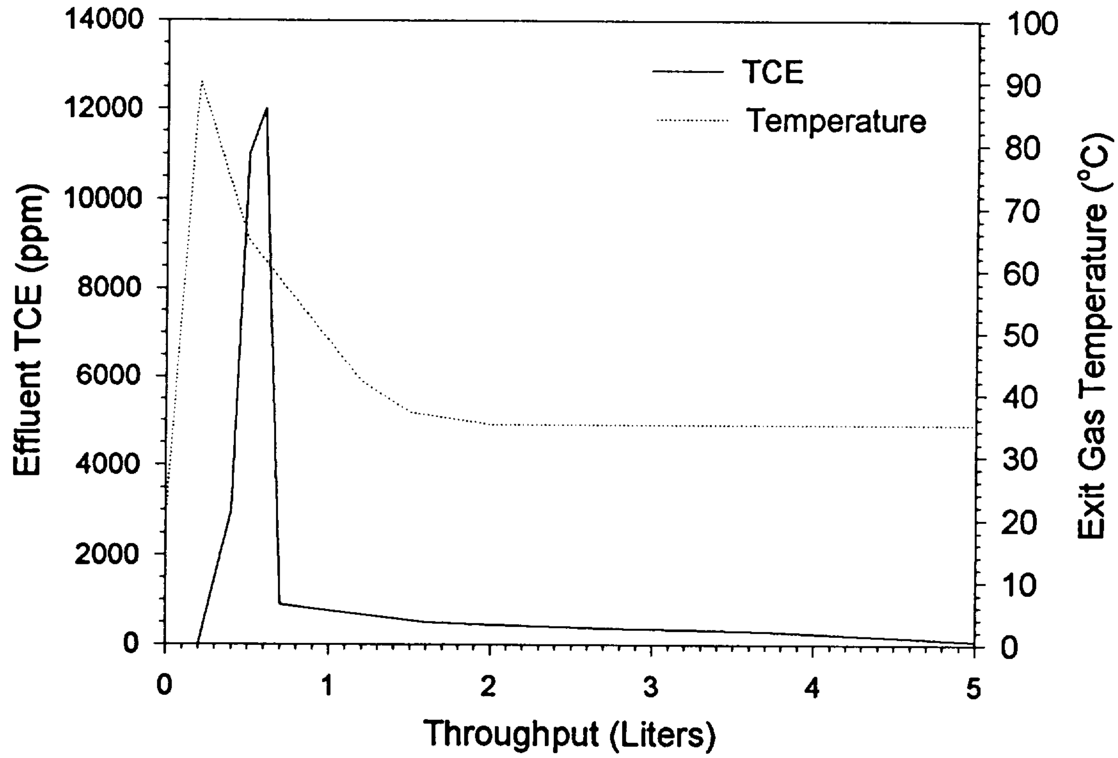


Figure 21. Thermal Regeneration of TCE Loaded ZSM-5.

1 L/min. At the same time full microwave power was applied. Maximum temperatures achieved were 90°C at 3 minutes into the regeneration. Afterward the temperature slowly decreased. These data are consistent with the interpretation that water molecules were the primary agent for absorption of microwaves. Once the water was removed from the system, temperature began to fall. To confirm that the silica gel bed was, adequately regenerated, a second sorption was conducted. The results, illustrated in Figure 23, indicated a complete restoration of the bed's sorption capacity.

Similar experiments were performed using a packed bed containing 3.47 cm^3 (2.12 g) of Molecular Sieve 13X. During sorption, exit gas temperatures of 62°C were attained. A four minute thermal desorption was performed under full microwave power. Exit gas

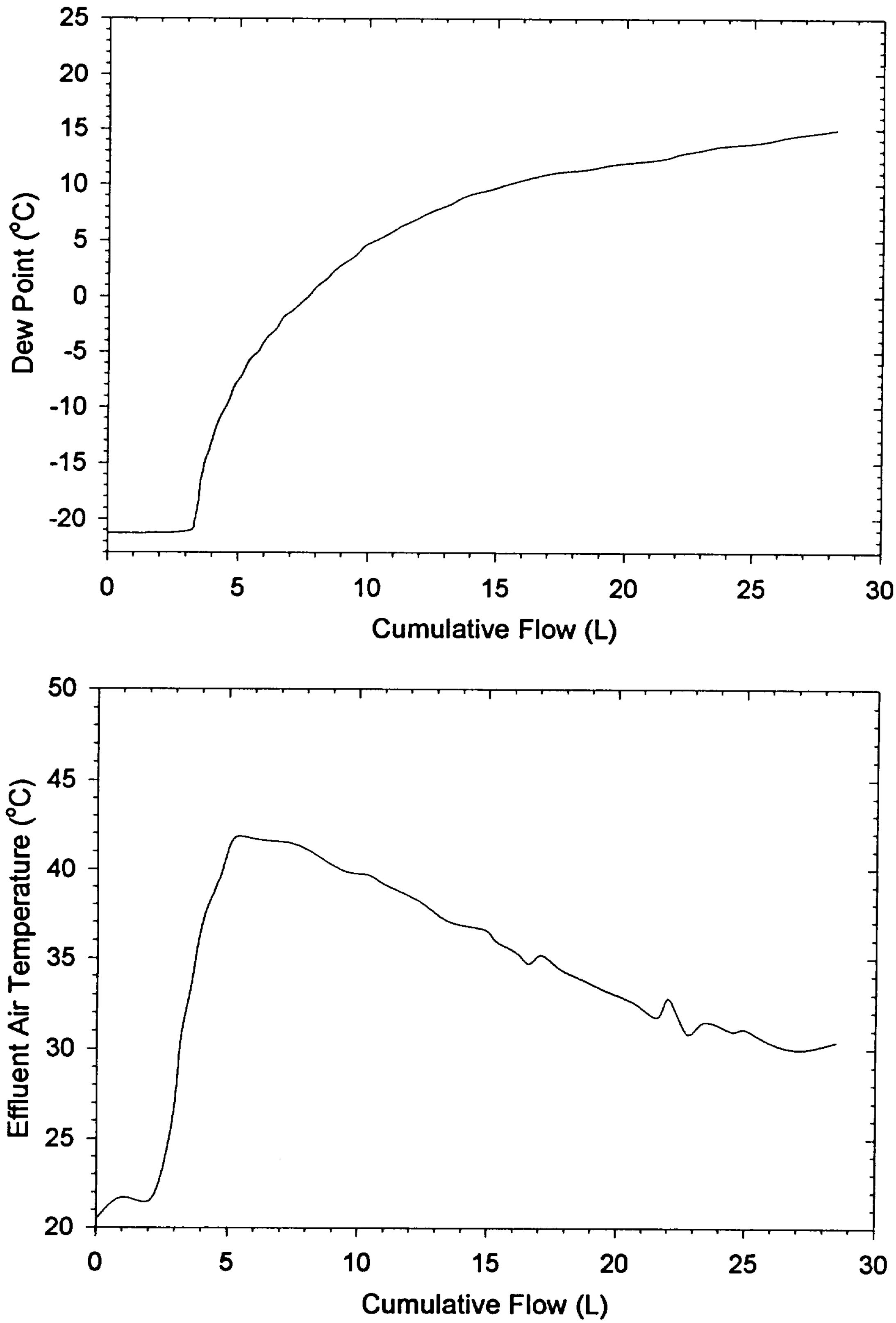


Figure 22. Initial Sorption of Water Vapor on Silica Gel.

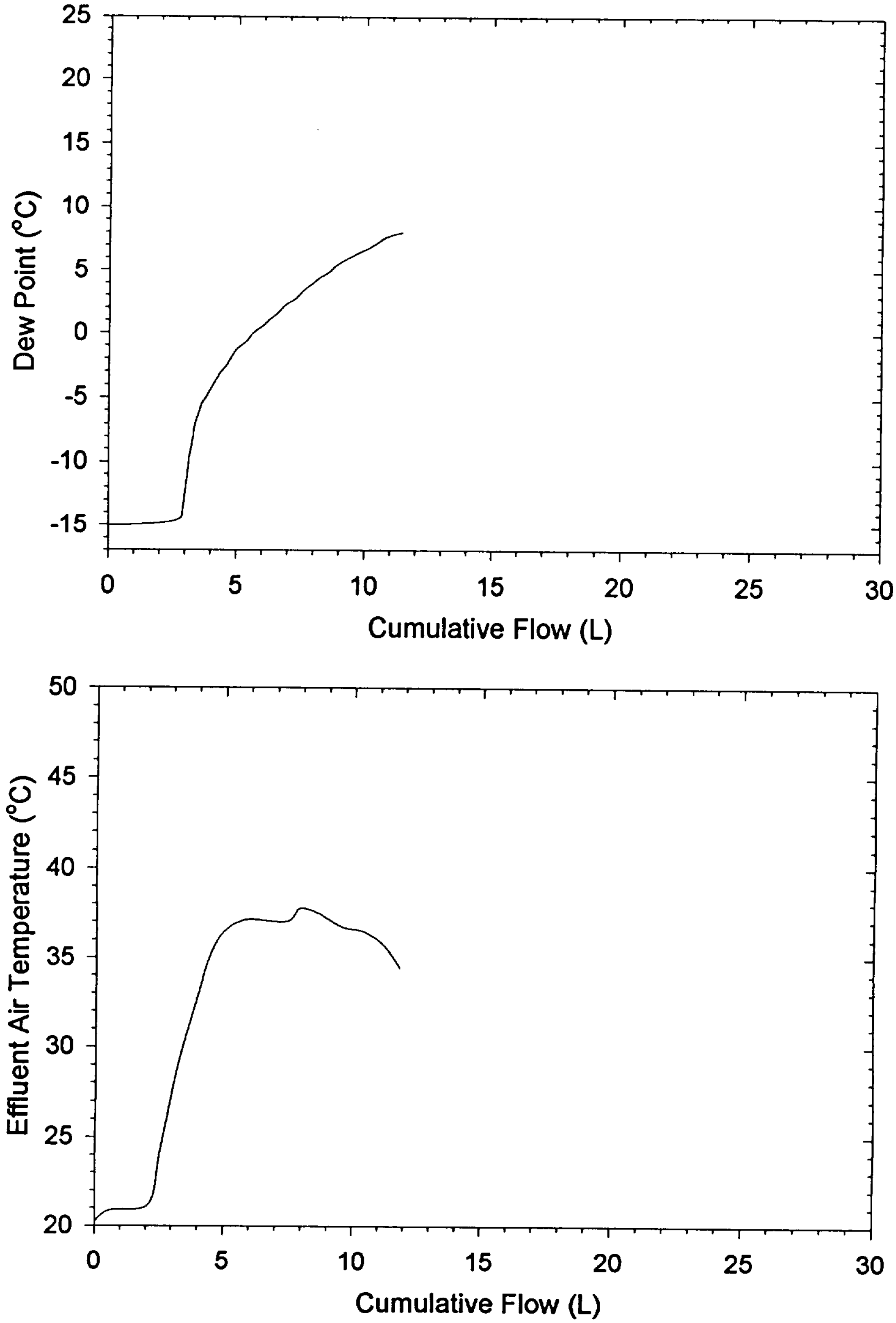


Figure 23. Sorption of Water Vapor on Silica Gel Following Regeneration.

temperature rose steadily throughout the regeneration, reaching 240°C after four minutes. The second sorption of water vapor indicated complete regeneration of the molecular sieve 13X. The initial sorption and second sorption are shown in Figures 24 and 25, respectively. The temperatures achieved during regeneration indicate fairly strong coupling of the molecular sieve with the microwave field. The experimental results obtained using a 5 cm^3 packed bed of Molecular Sieve 5A are given in Figures 26-28. During sorption maximum exit gas temperatures of 52°C were observed. Under full microwave power during desorption, exit gas temperatures rose to 118°C until irradiation was terminated 14 minutes into the regeneration.

Based upon these data, it appears that silica gel couples only very weakly with the incident microwave energy, and that regeneration is in large part attributable to the direct uptake of energy by adsorbed water molecules. On the other hand, Molecular Sieve 13X appears to be quite susceptible to microwave heating. An intermediate case is presented by Molecular Sieve 5A, for which significant microwave absorption by the solid medium and by adsorbed water appears to occur.

Sorption and Regeneration of Layered Media for CO_2, Water Vapor and Trace Organics.

A layered composite sorbent bed consisting of 0.56 g Molecular Sieve 13X, 0.17 g Carbosieve S-III, and 2.27 g Molecular Sieve 5A, in sequence from inflow face to outlet, was prepared for a challenge with an air stream saturated with water vapor and containing 0.5% CO_2, 628 ppm acetone, and 105 ppm TCE. Sorption was conducted at a flow rate of 100 mL/min. Effluent levels of CO_2, total hydrocarbon, and dew point were monitored continuously. Carbon dioxide began initial breakthrough at 5.2 liters of cumulative throughput. Fifty percent breakthrough occurred at approximately 9.5 liters, and total breakthrough was observed at 14 liters. Hydrocarbon breakthrough began after 9.2 liters of cumulative flow and never reached 50%. Water adsorption as indicated by dew point was anomalous. The dew point fell steadily until the 9 liter mark had been reached, after which it

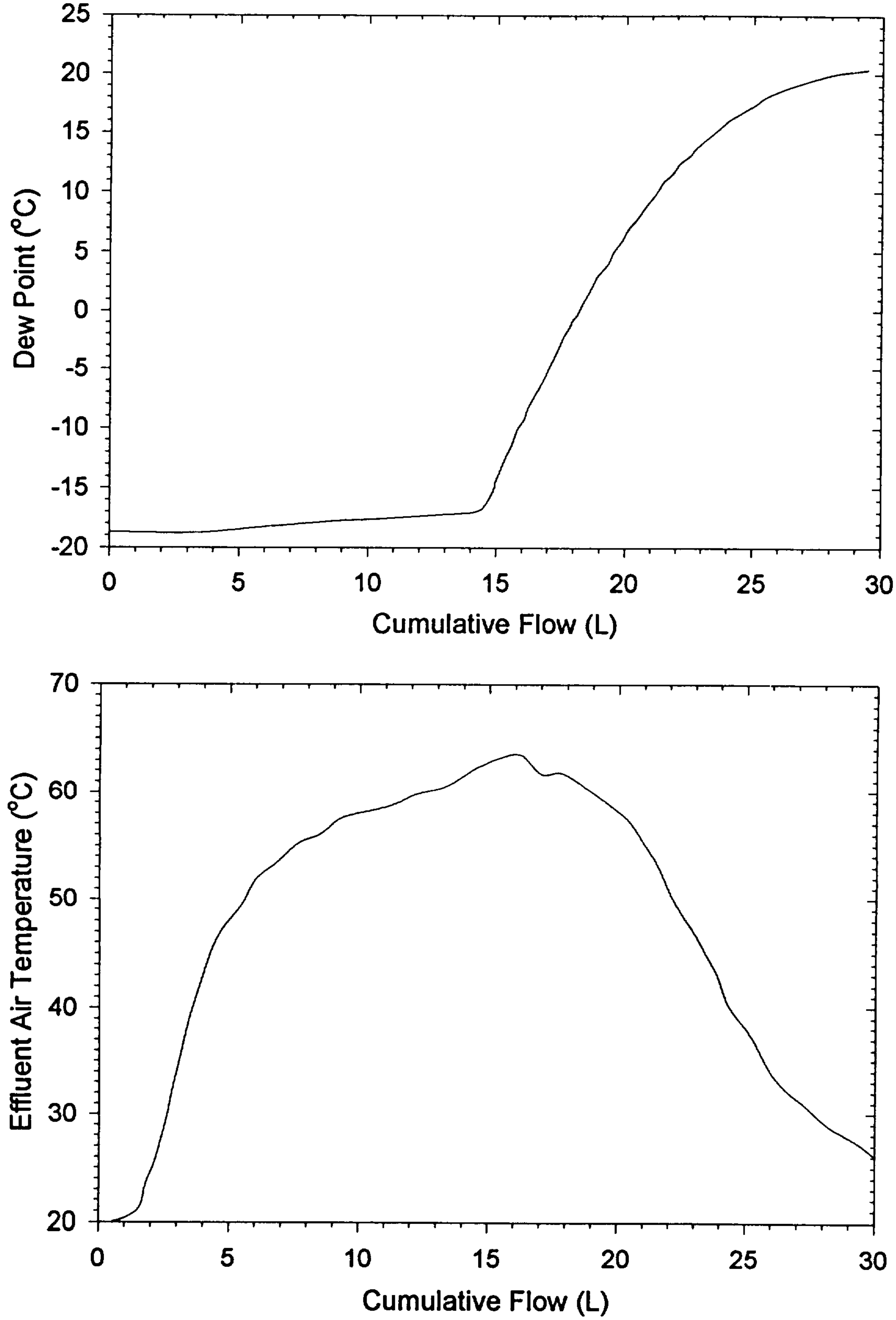


Figure 24. Initial Sorption of Water Vapor on Molecular Sieve 13X.

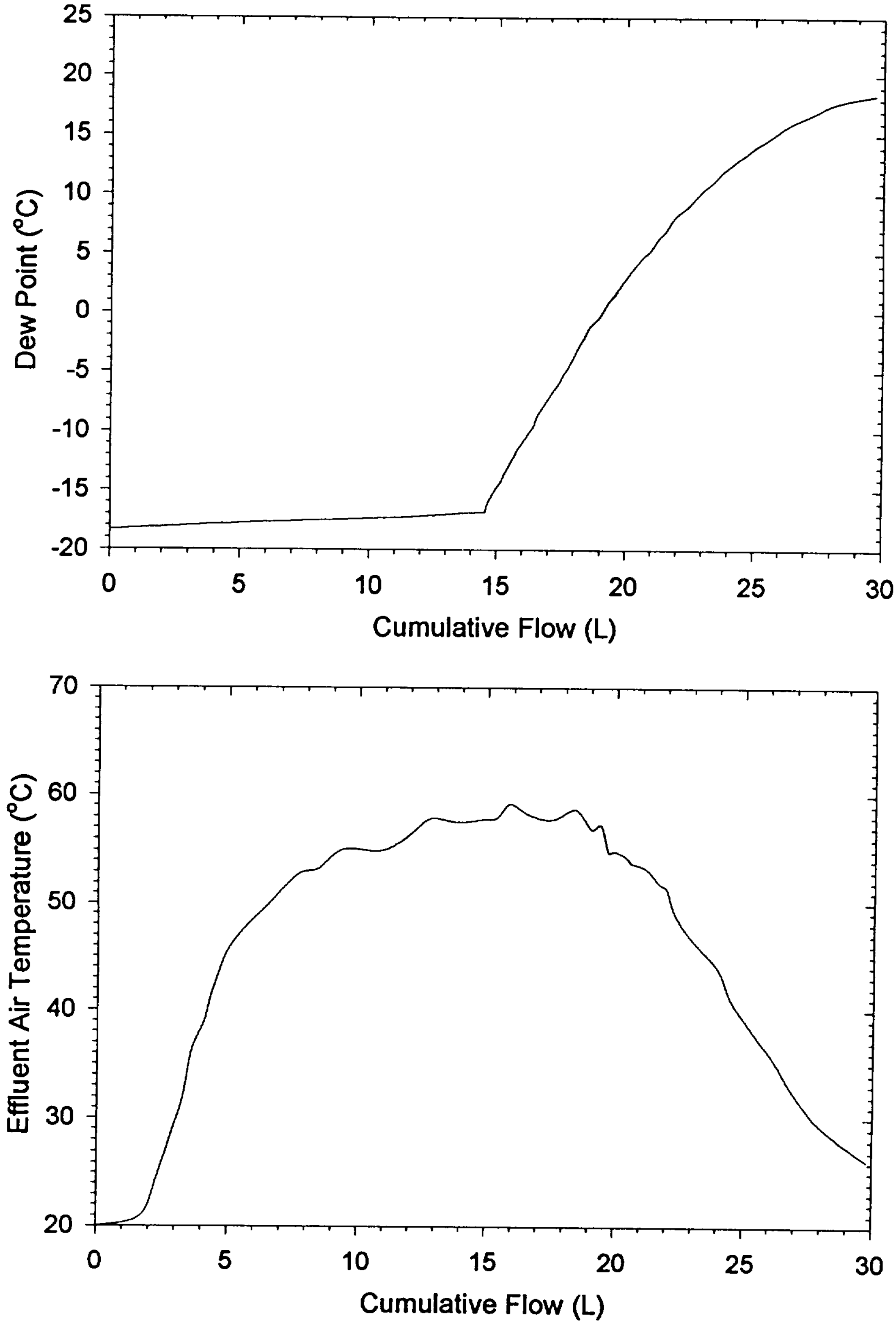
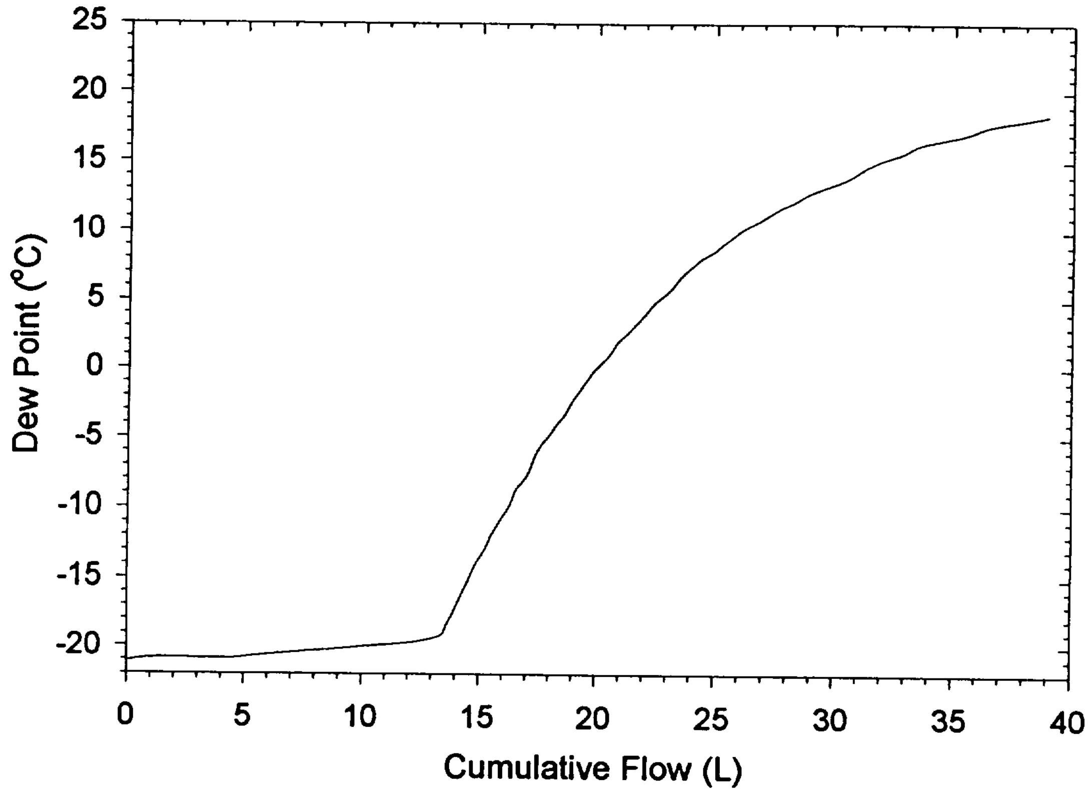


Figure 25. Sorption of Water Vapor on Molecular Sieve 13X Following Regeneration.



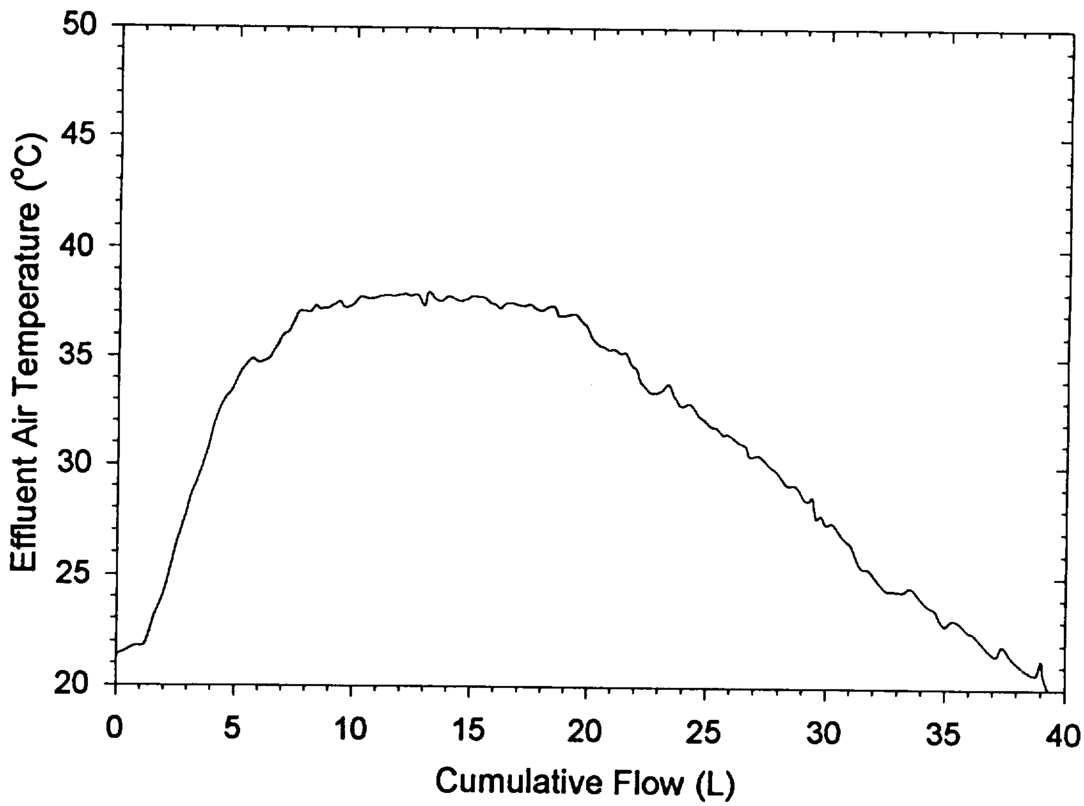


Figure 26. Initial Sorption of Water Vapor on Molecular Sieve 5A.

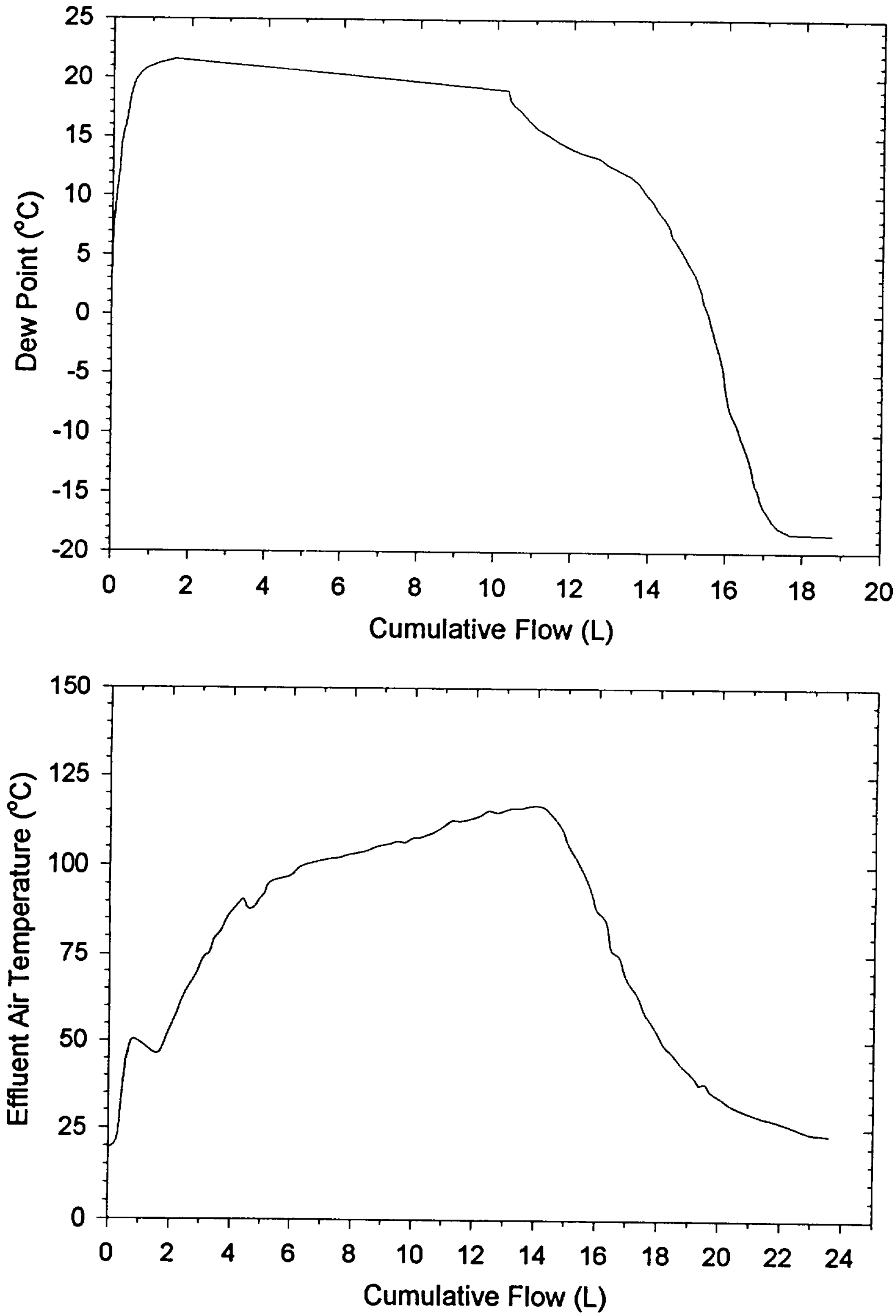
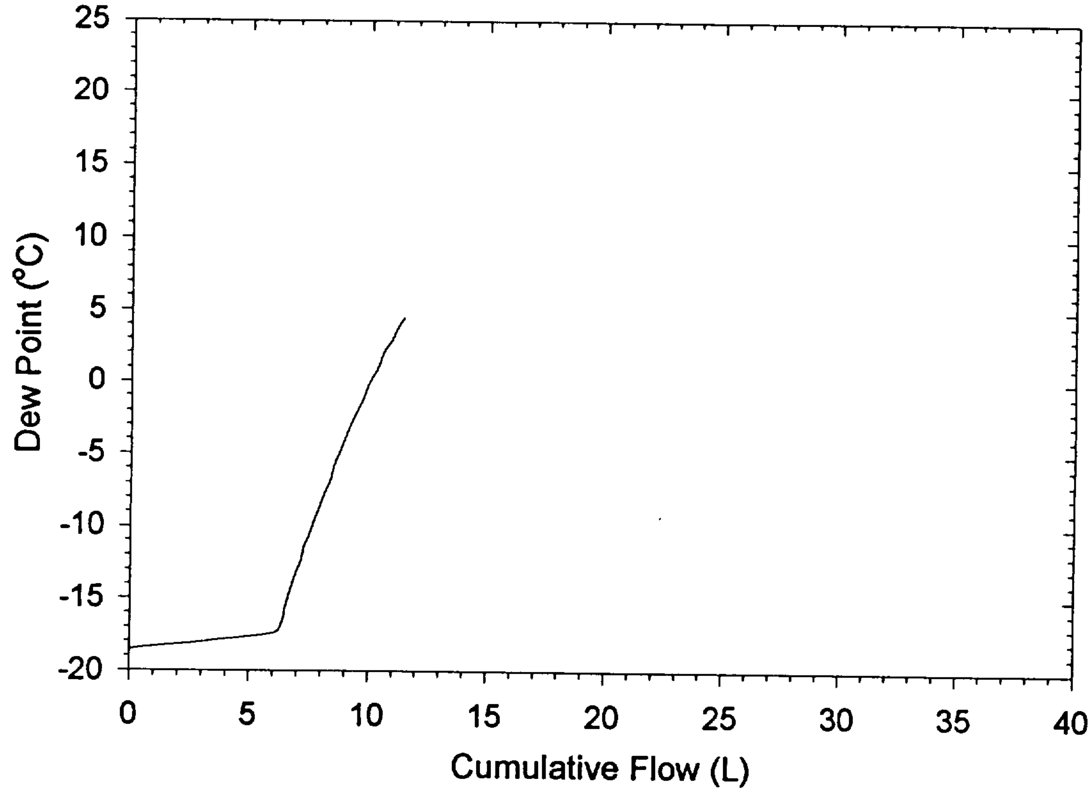


Figure 27. Thermal Regeneration of Water Loaded Molecular Sieve 5A.



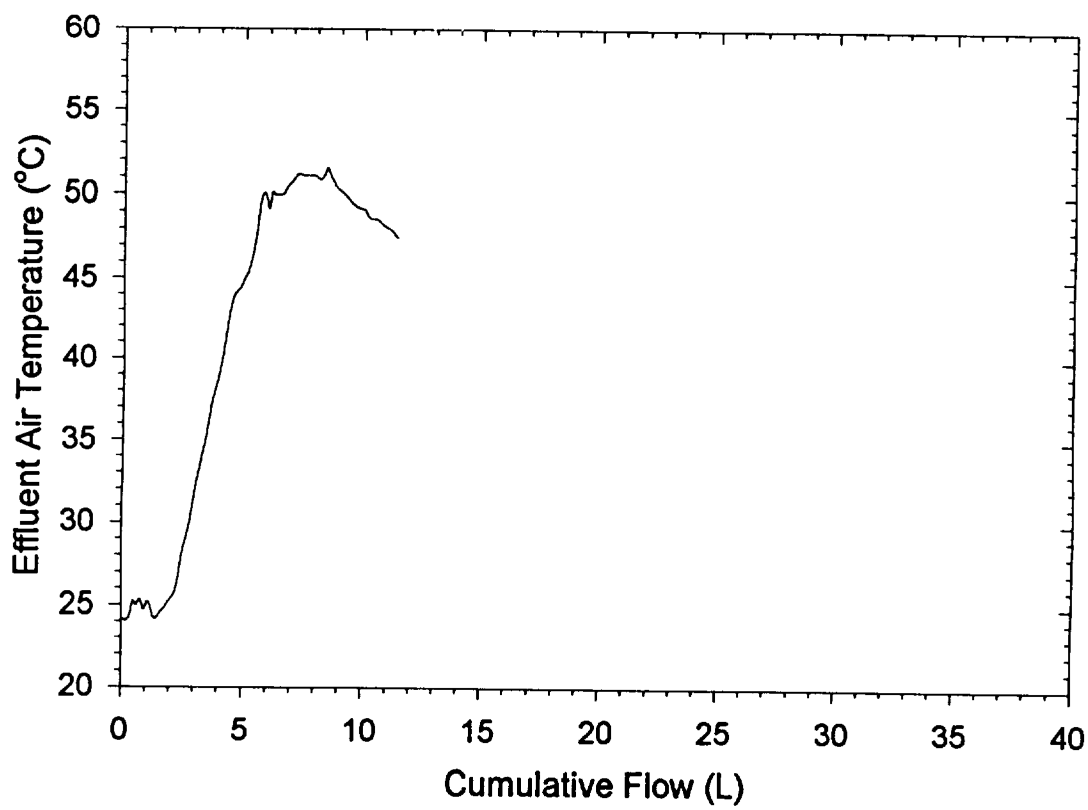


Figure 28. Sorption of Water Vapor on Molecular Sieve 5A Following Regeneration.

began to rise. Because the dew point never exceeded -10°C, adequate removal of water vapor by the layered composite bed was indicated.

Based upon 100% breakthrough of CO_2, regeneration was initiated after 14 liters of flow. Maximum effluent gas temperatures of approximately 45°C were observed during regeneration. Following regeneration, a second sorption was conducted, yielding results which were essentially identical with those obtained initially. This confirmed the adequacy of the microwave powered thermal regeneration. The results of the initial and second sorption experiments are shown in Figures 29, and 30 respectively.

Microwave Reflection and Transmission Spectra.

All of the microwave powered thermal regenerations attempted in the current study utilized 2.45 GHz as the frequency of irradiation. To evaluate the potential for more favorable microwave heating of sorbent materials using other frequencies, a spectral study of microwave transmission, reflection, and phase shift over the frequency range between 1.3 - 2.7 GHz was conducted using the vector network analyzer based multifrequency microwave transmission and reflection apparatus. Transmitted and reflected power levels were measured in terms of decibels (dB), defined as,

$$\mathrm{dB} = 10 \log \frac{P_m}{P_r}$$

where P_m is the measured power level and P_r is a reference power level, in this case a 20 dB sample of the network analyzer RF output.

Transmission and reflection spectra are presented for the empty sample chamber, 830IMC activated carbon, #5769 activated carbon, lithium hydroxide, Molecular Sieve 5A, Molecular Sieve 13X, dry silica gel, wet silica gel, and silver carbonate in Figures 31-39 respectively. The phase shifts of transmitted power for these materials as a function of frequency are presented in Figures 40-44.

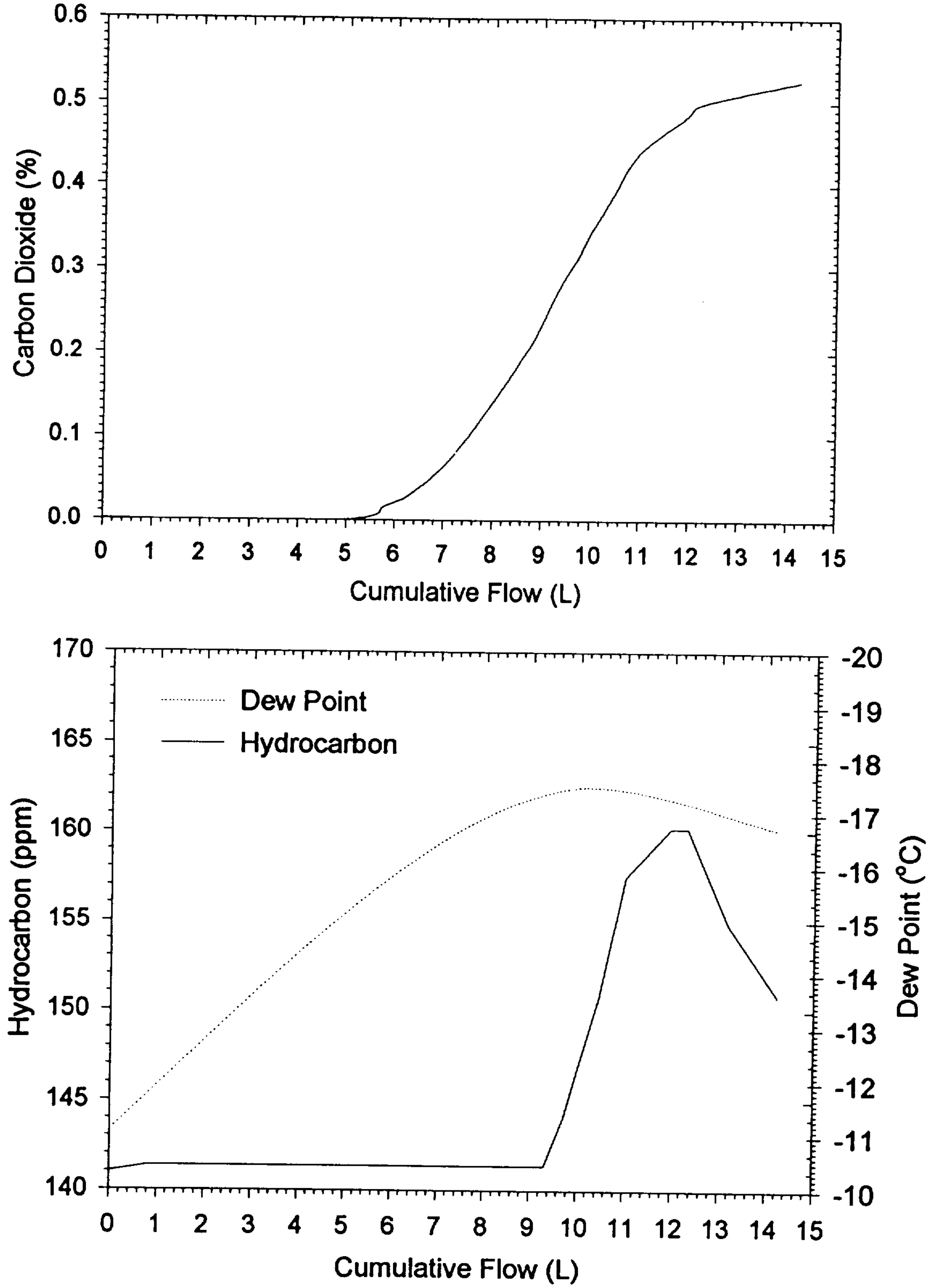


Figure 29. Composite Bed: Initial Sorption of CO_2, Water Vapor, Acetone and TCE.

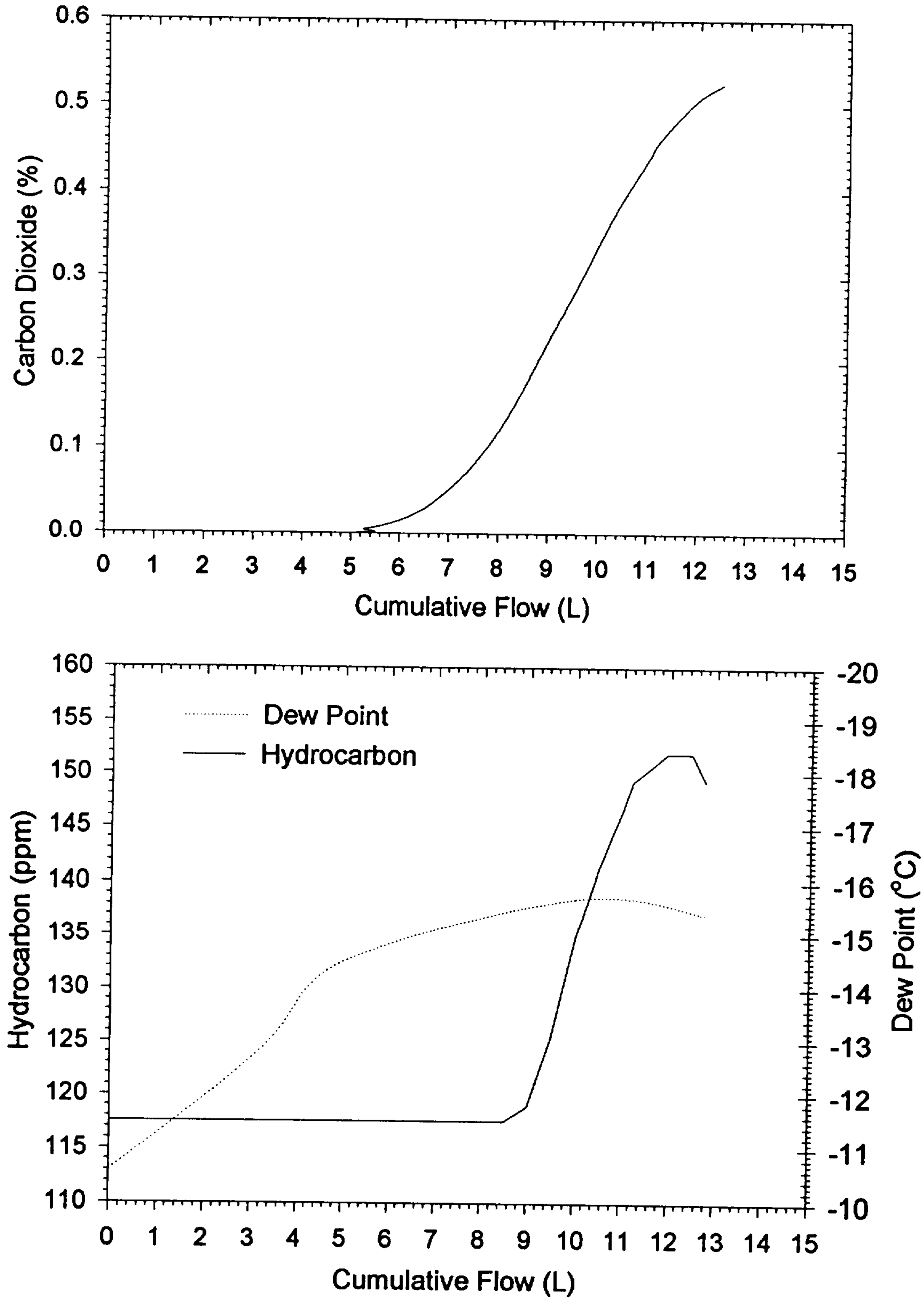


Figure 30. Composite Bed: Repeat Sorption after Microwave Powered Regeneration..

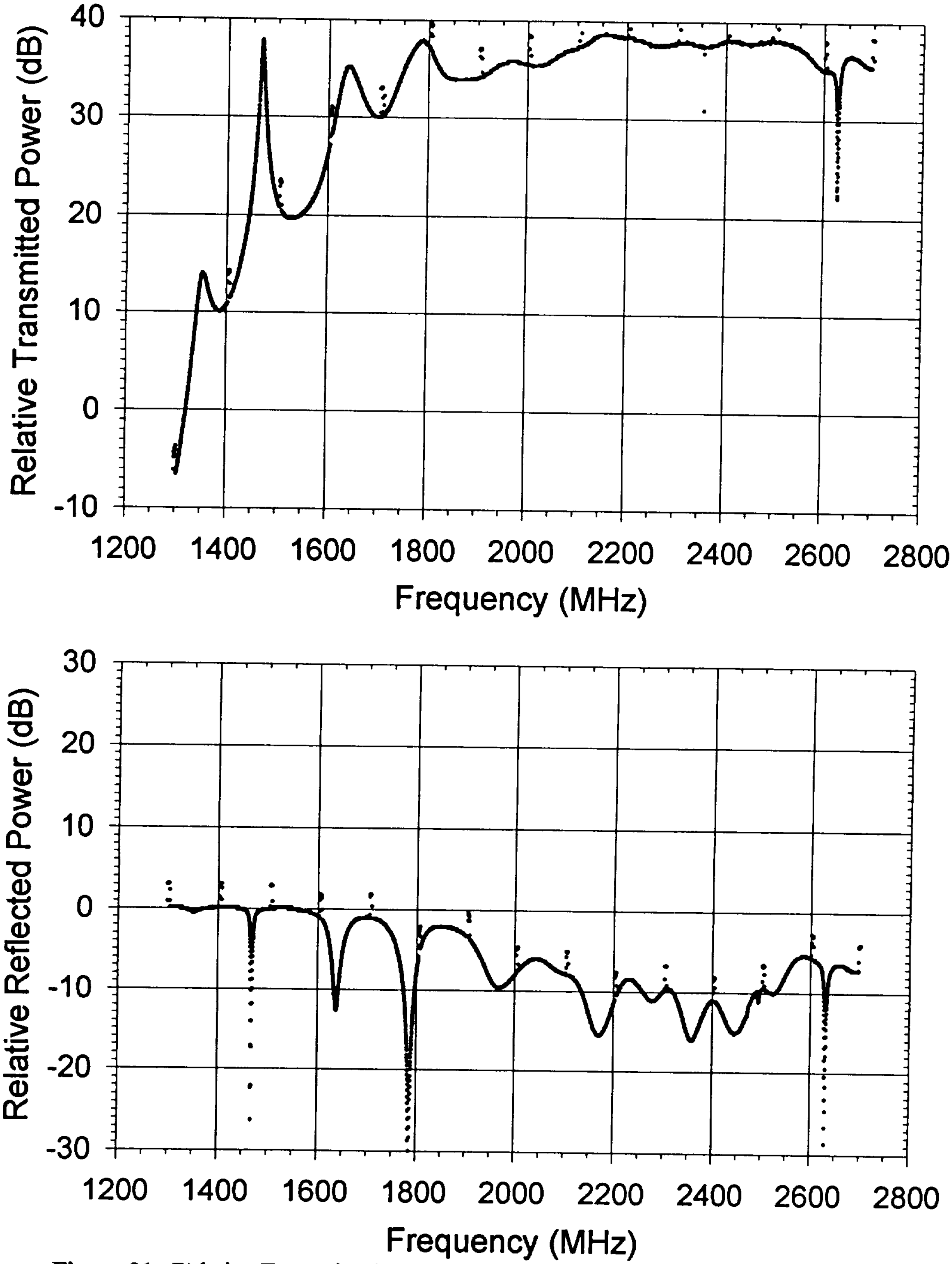
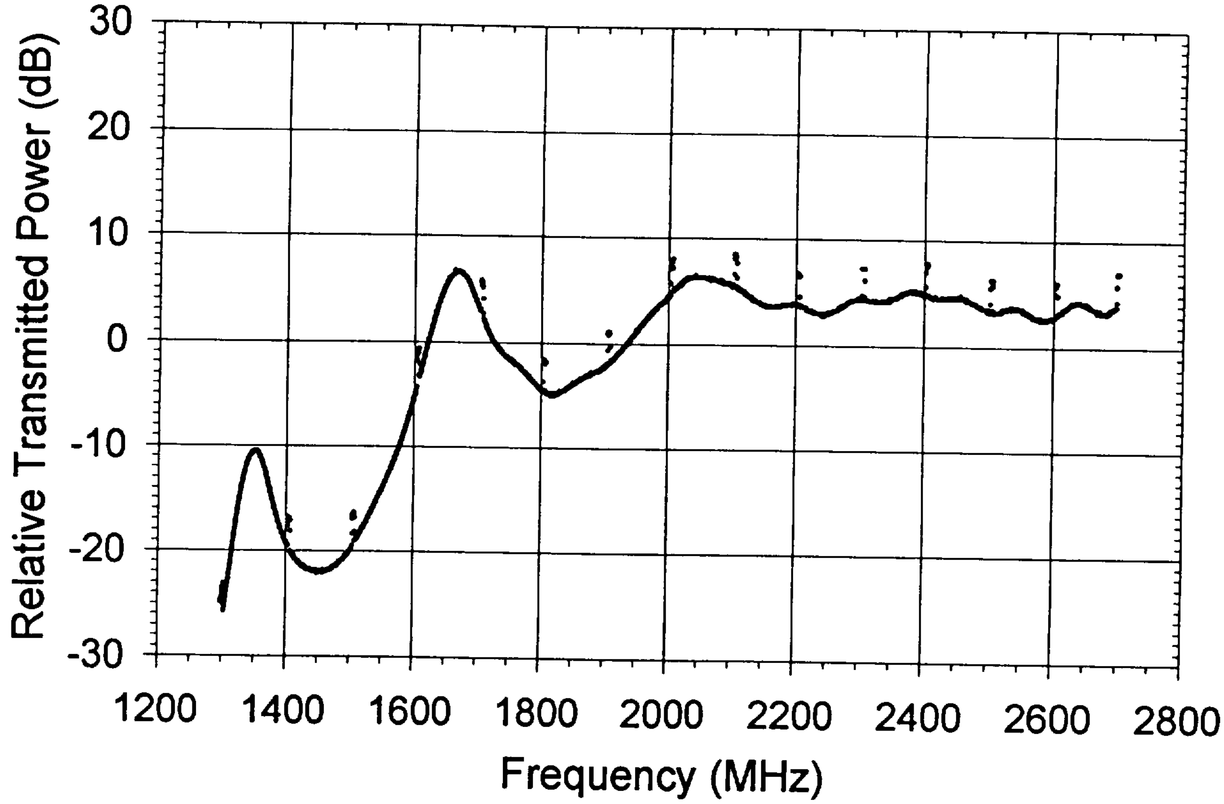


Figure 31. Relative Transmitted and Reflected Power for Empty Sample Chamber.



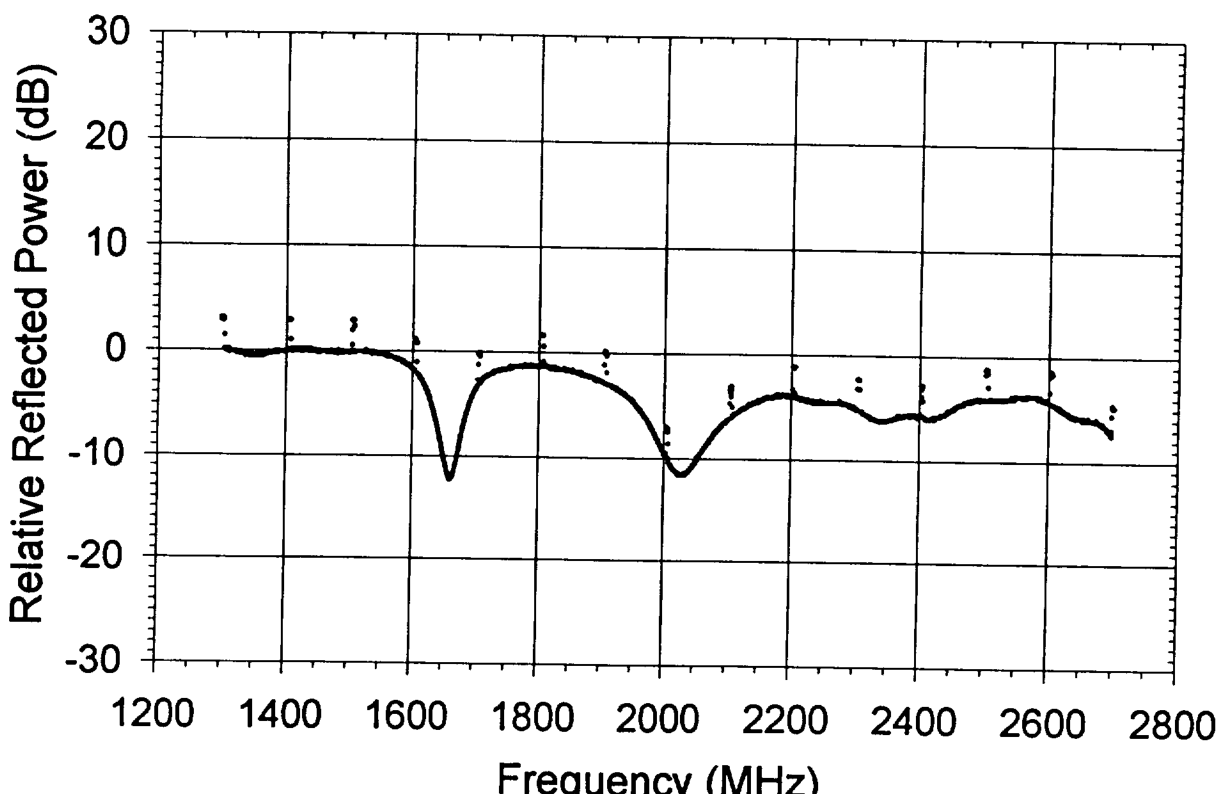


Figure 32. Relative Transmitted and Reflected Power: CECA-830IMC Activated Carbon.

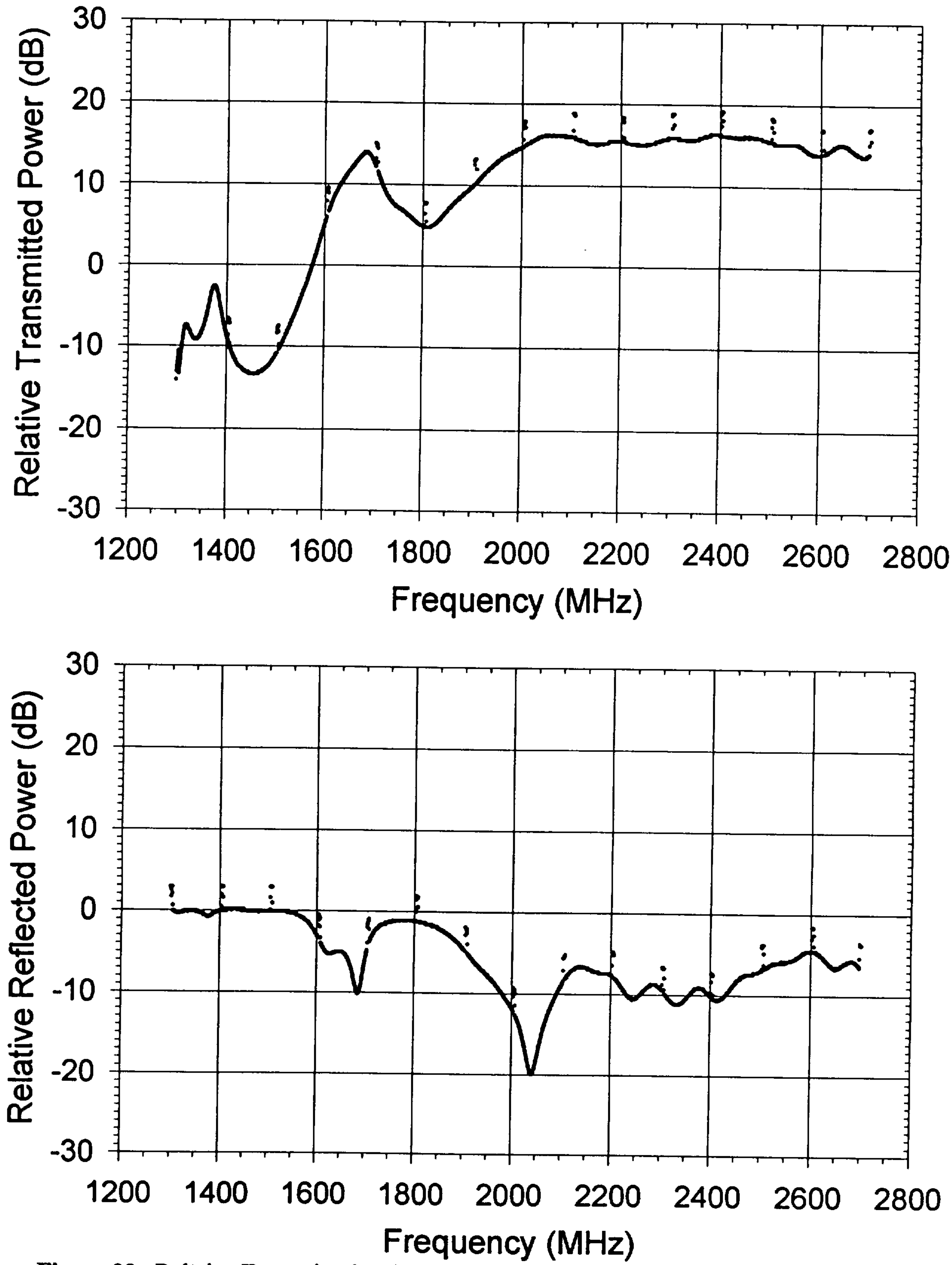
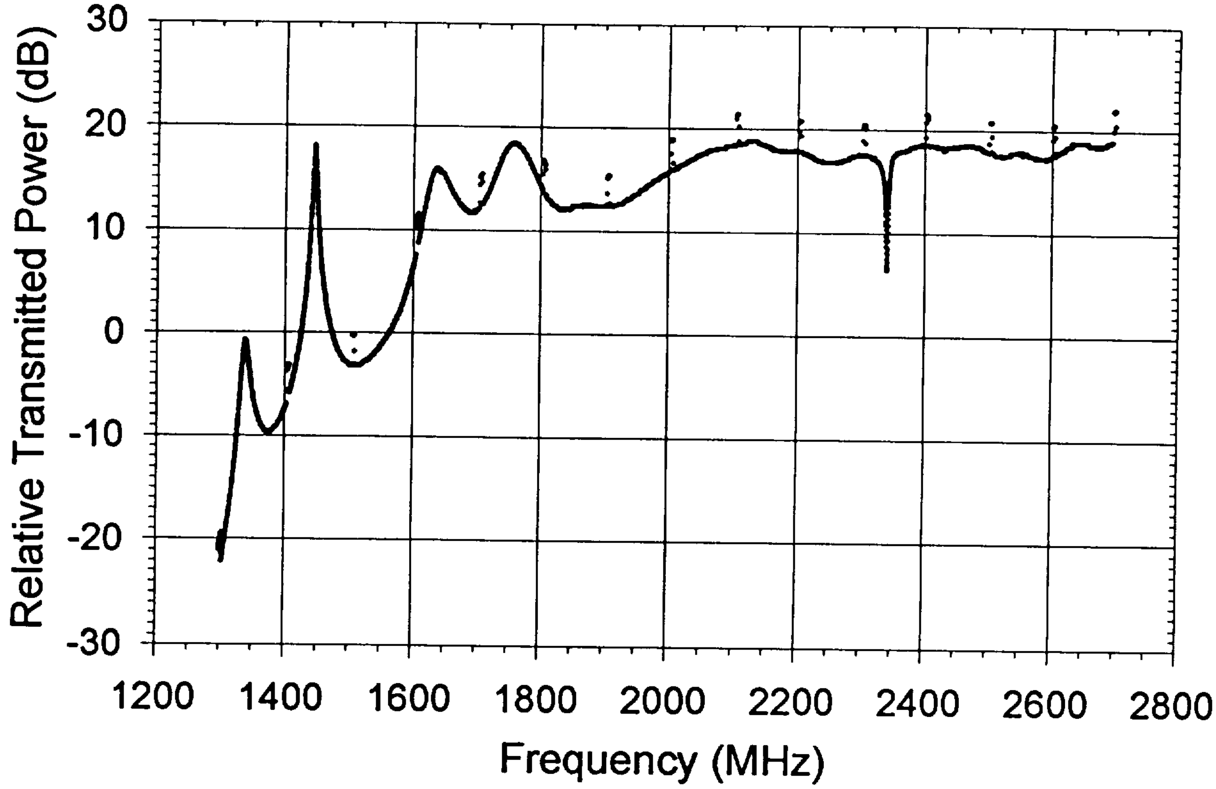


Figure 33. Relative Transmitted and Reflected Power: Alltech 5769 Activated Carbon.



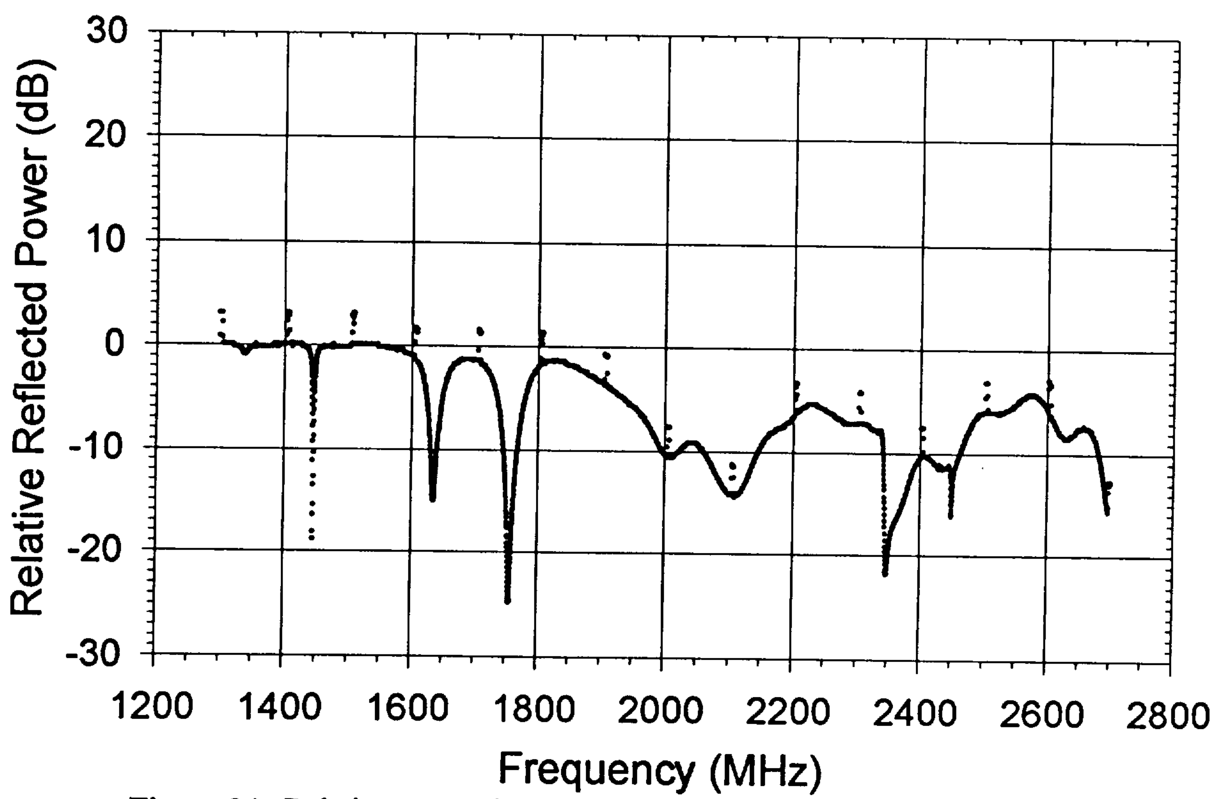


Figure 34. Relative Transmitted and Reflected Power: Lithium Hydroxide

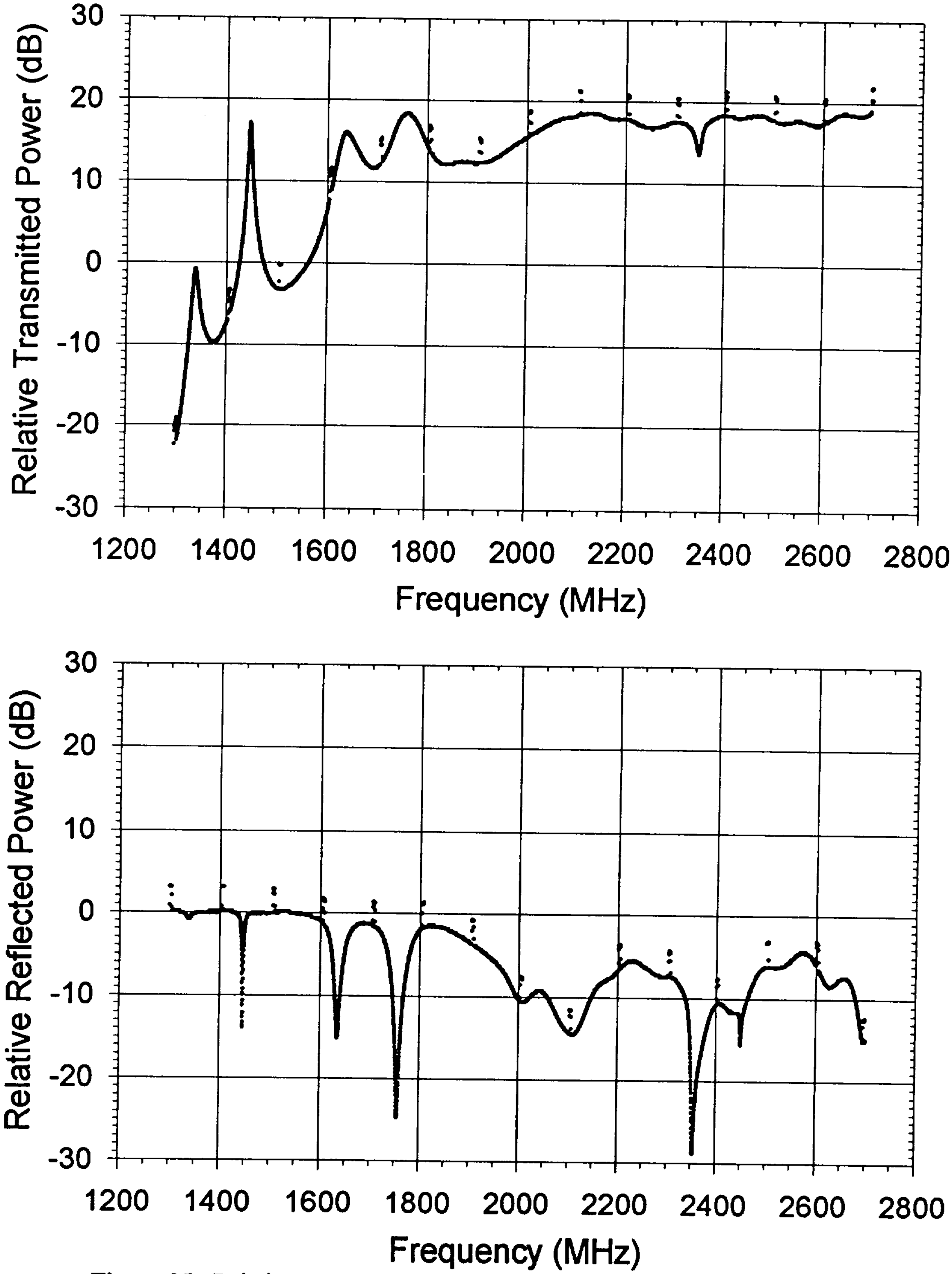


Figure 35. Relative Transmitted and Reflected Power: Molecular Sieve 5A.

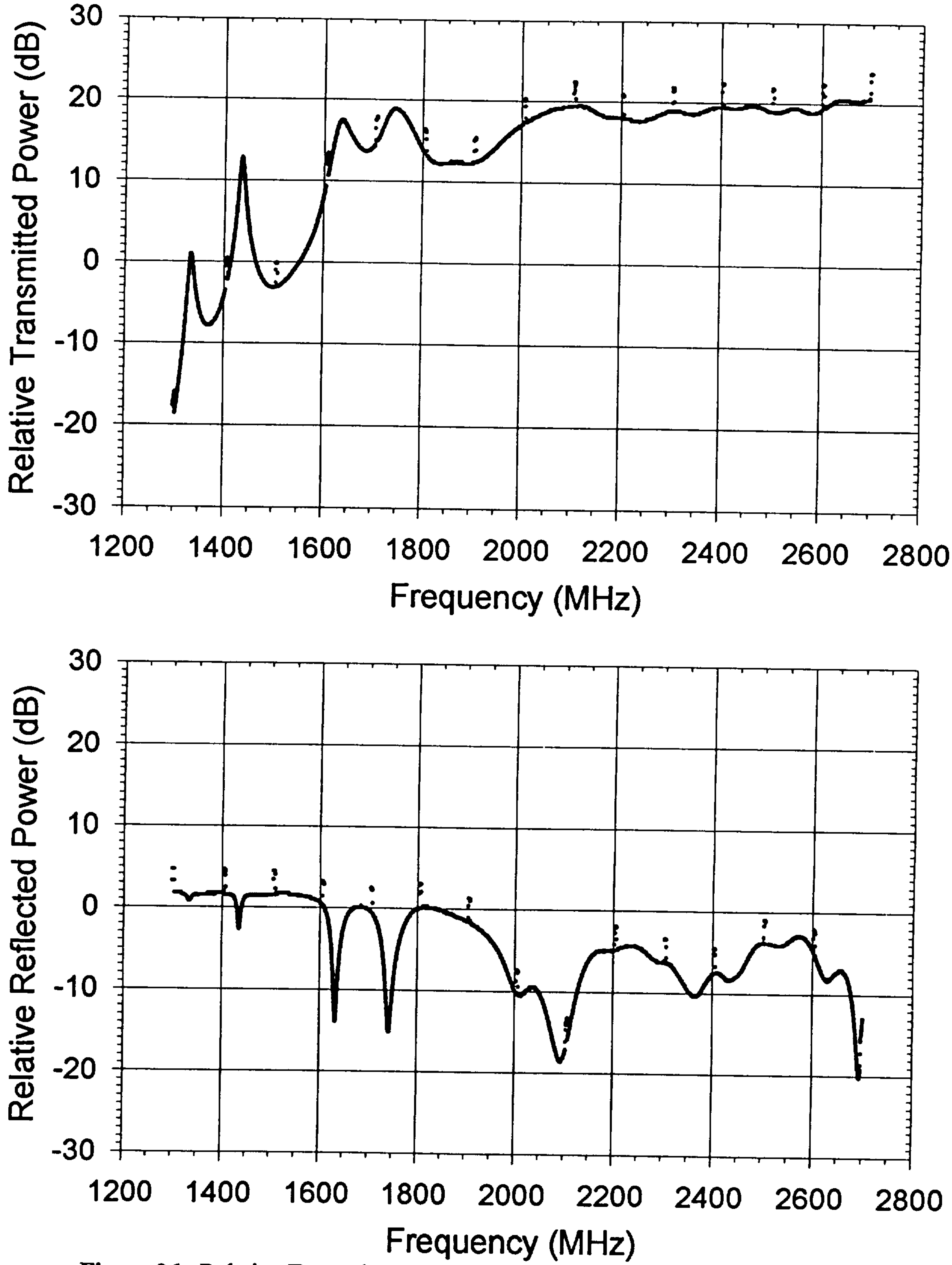
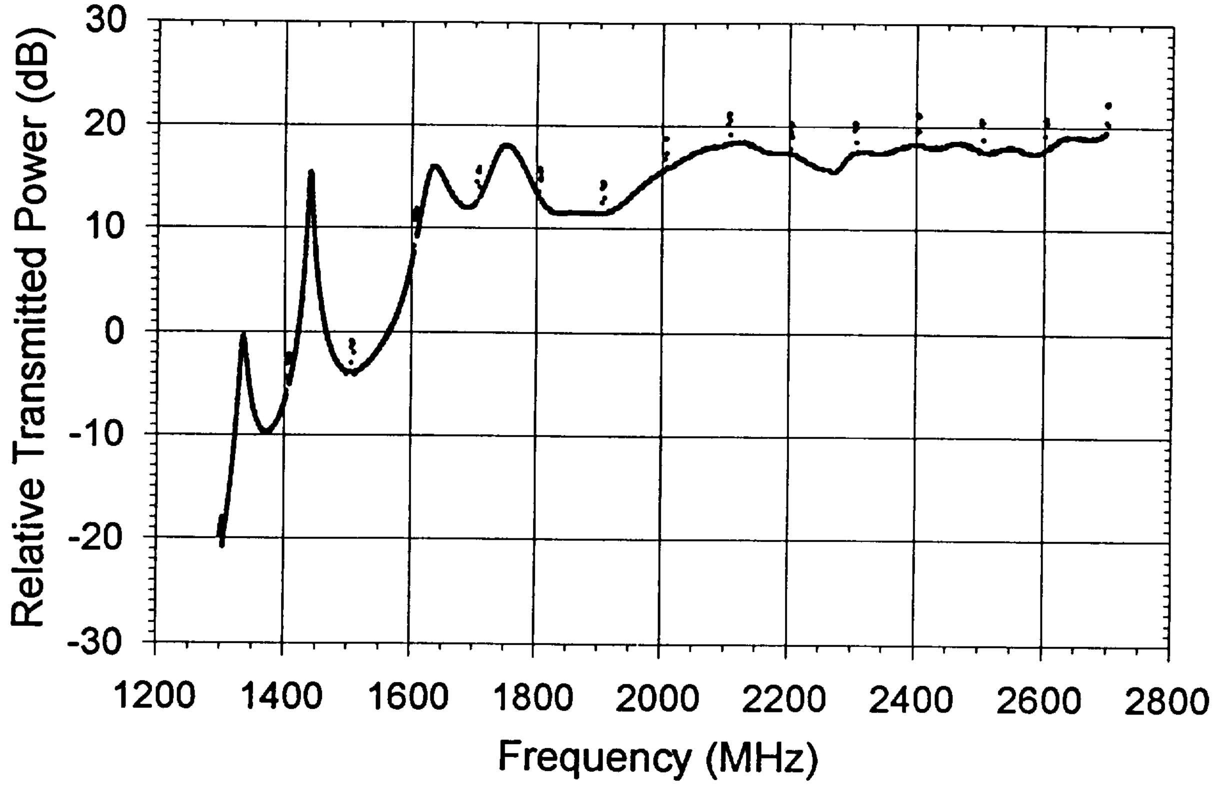


Figure 36. Relative Transmitted and Reflected Power: Molecular Sieve 13X.



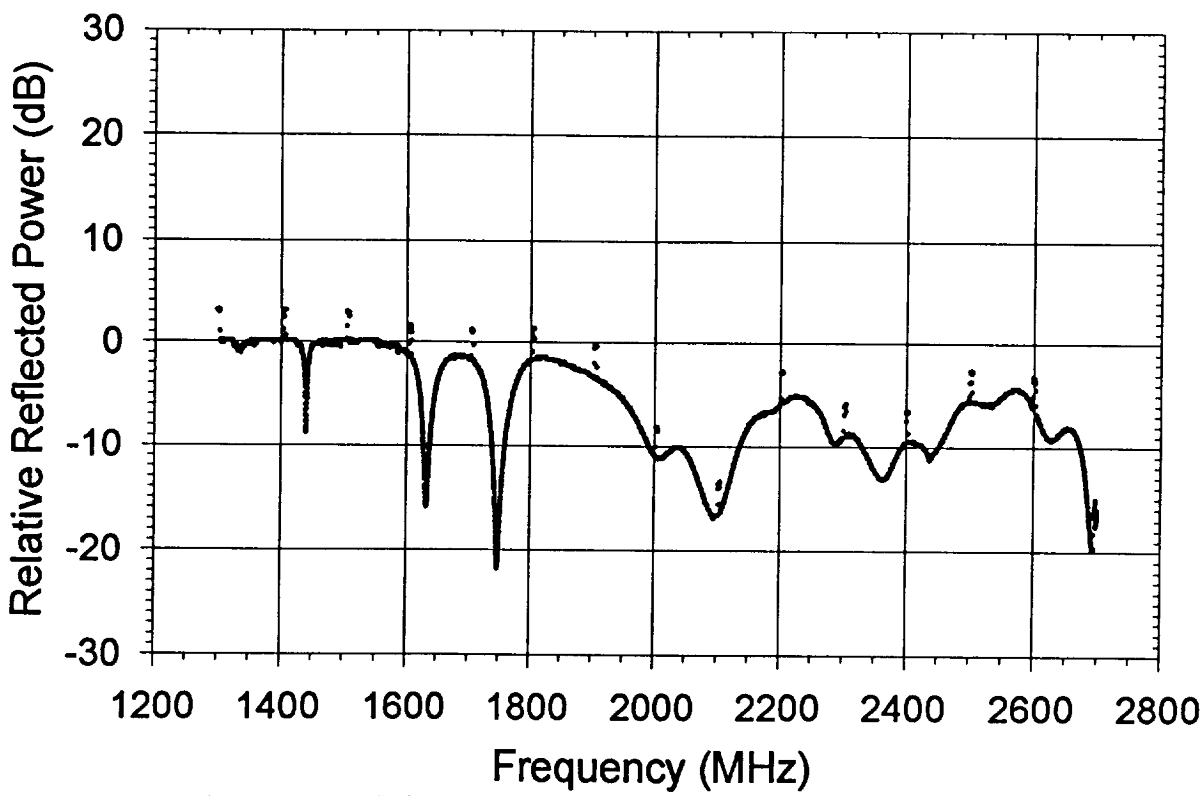


Figure 37. Relative Transmitted and Reflected Power: Silica Gel.

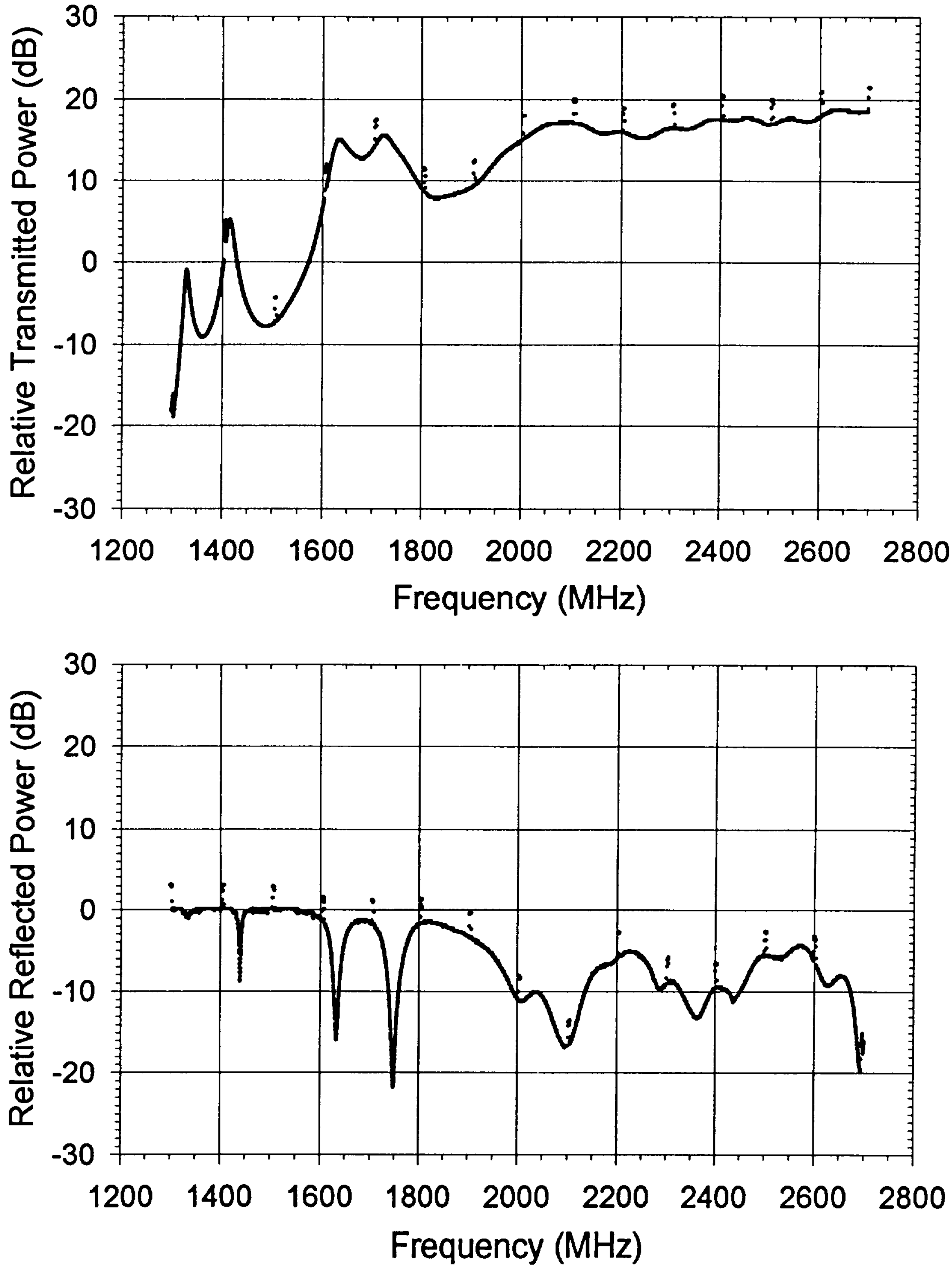


Figure 38. Relative Transmitted and Reflected Power: Silica Gel with Adsorbed Water.

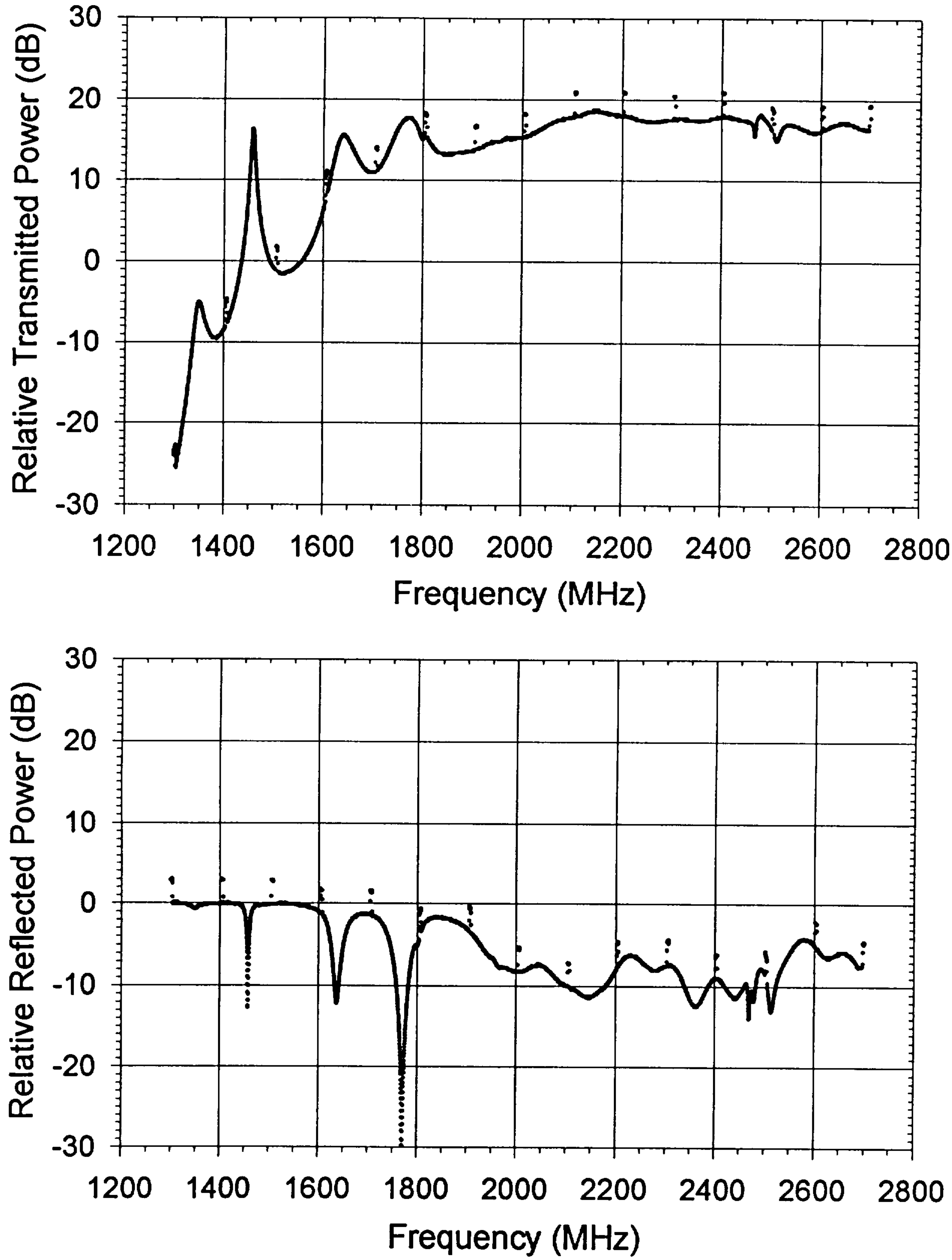
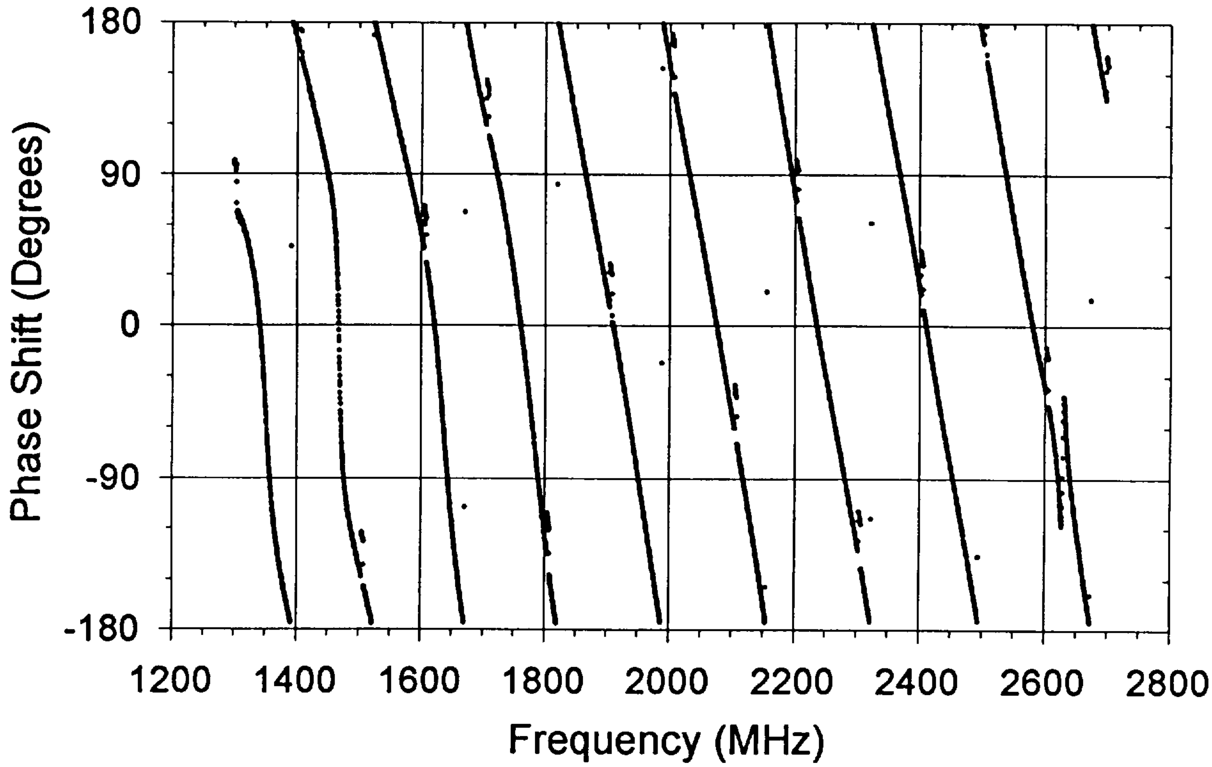


Figure 39. Relative Transmitted and Reflected Power: Silver Carbonate.



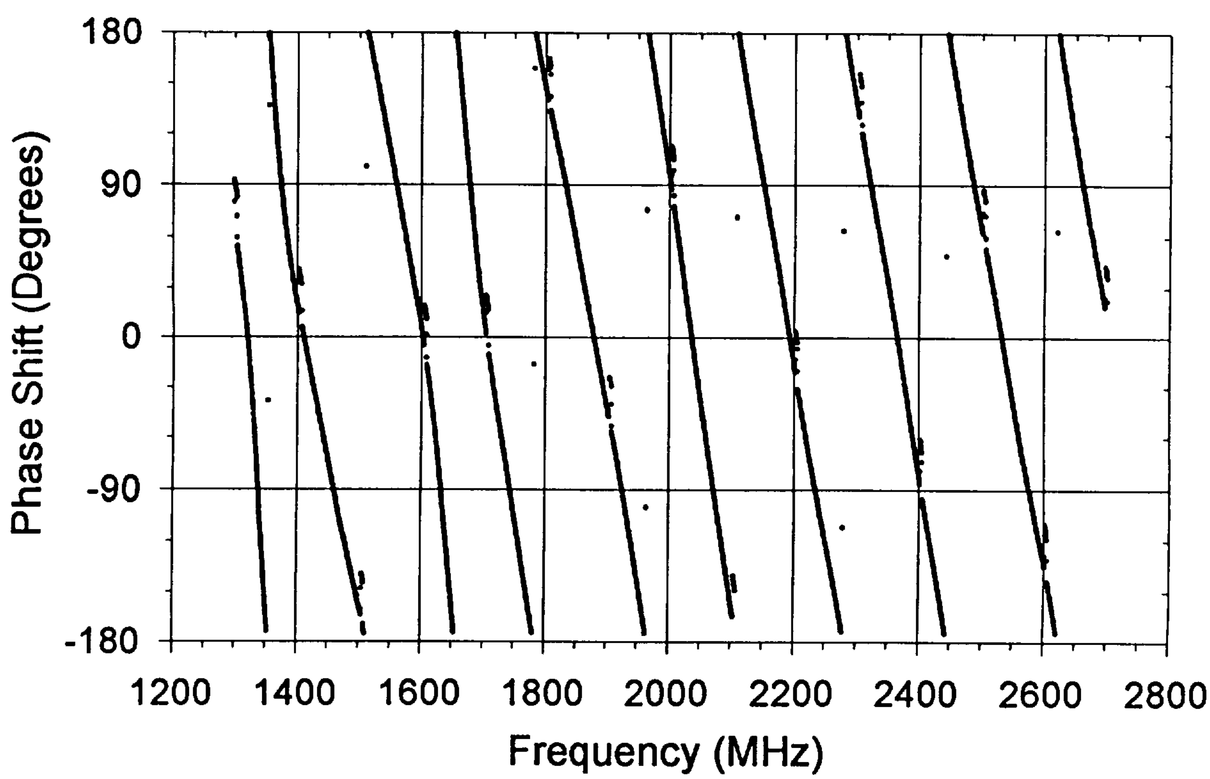


Figure 40. Transmitted Power Phase Shift: Top-Empty Cell, Bottom-CECA-830IMC.

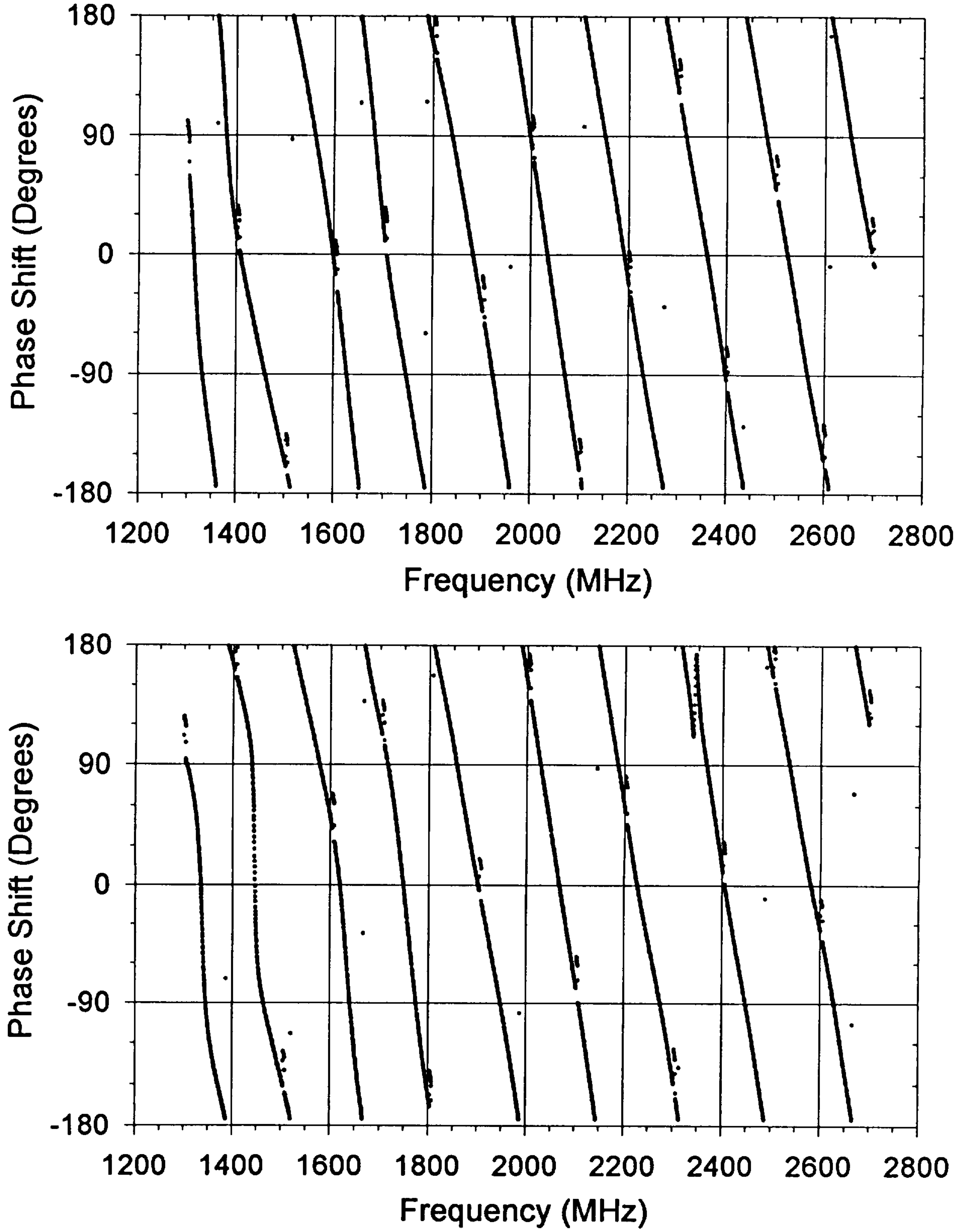


Figure 41. Transmitted Power Phase Shift: Top-5769 Activated Carbon, Bottom: LiOH.

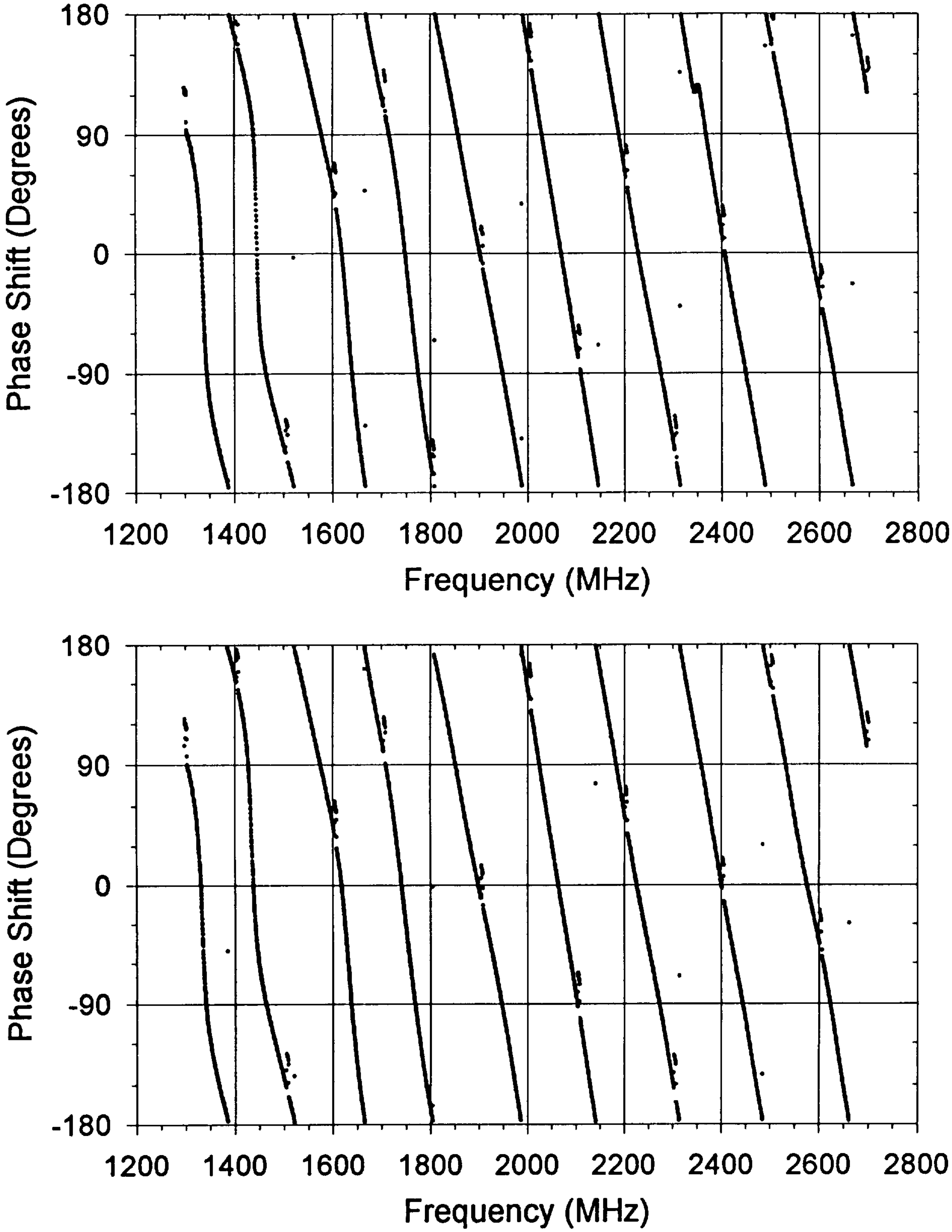


Figure 42. Transmitted Power Phase Shift: Top - MS 5A, Bottom - MS 13X.

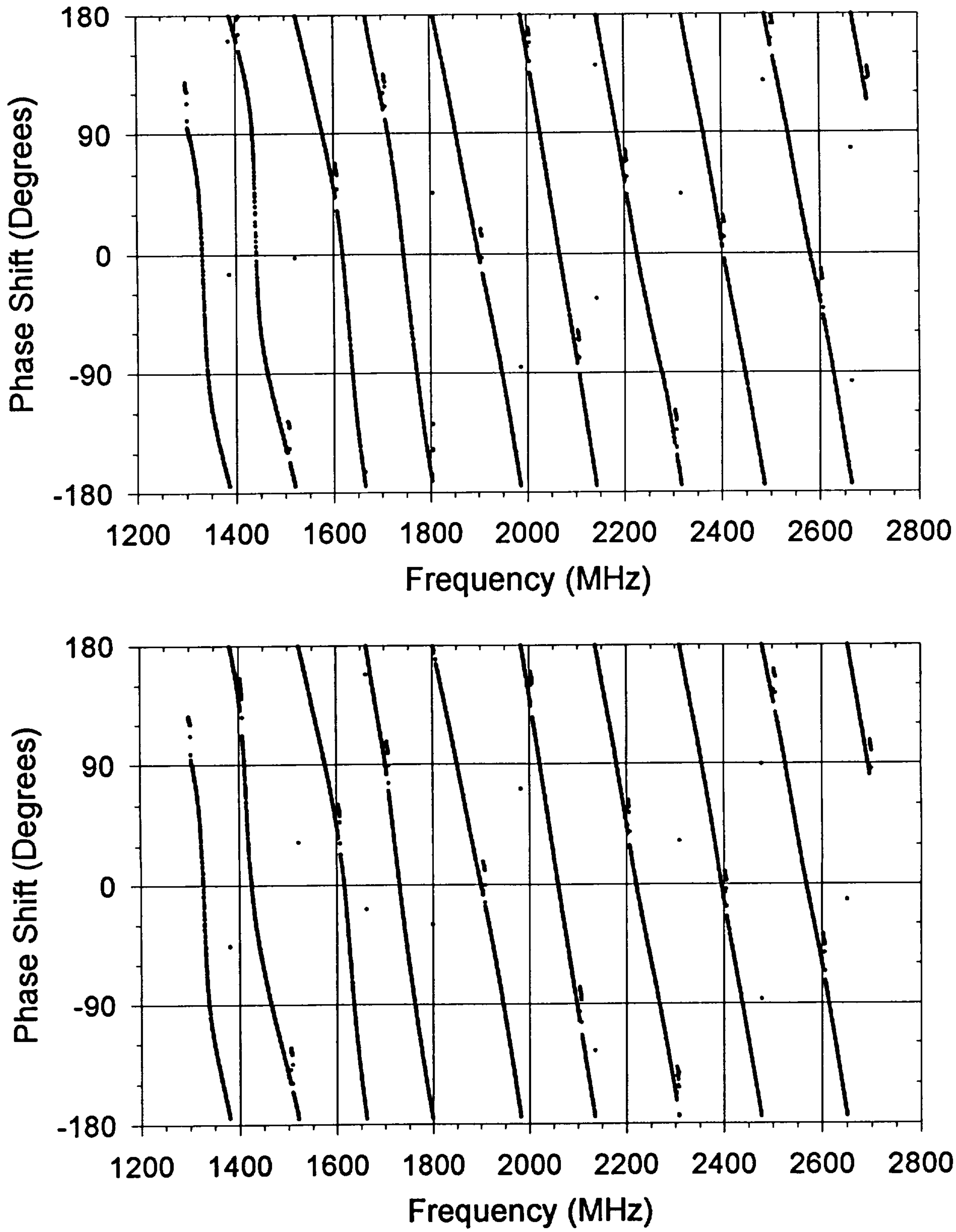


Figure 43. Transmitted Power Phase Shift: Top-Dry Silica Gel, Bottom-Wet Silica Gel.

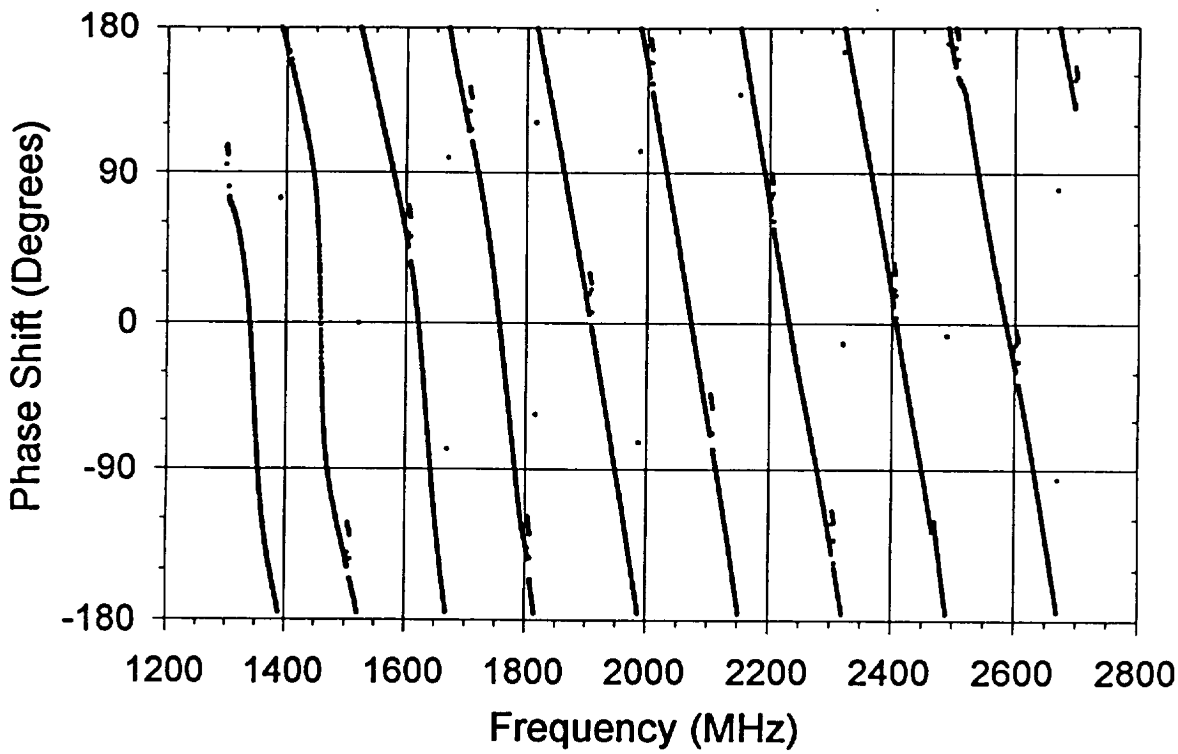


Figure 44. Transmitted Power Phase Shift: Silver Carbonate.

As a basis for comparison, these materials were also heated at 2.45 GHz in a rectangular waveguide system for 20 seconds. The samples were enclosed within glass Petri dishes. At the end of the microwave exposure, the waveguide was opened and the temperature of the media were determined using an IR thermometer. The results are presented in Table II.

Table II. Waveguide Microwave Heating Tests Results at 2.45 GHz.

Sorbent	**Mass (g)**	**ΔT (°C)**
Empty Petri Dish	-	1
Activated Carbon - IMC830	4.34	> 204 (off scale)
Activated Carbon - 5769	4.22	218
Lithium Hydroxide	5.98	0
Molecular Sieve 5A	5.90	5
Molecular Sieve 13X	9.26	38
Silica Gel	5.6	17
Silver Carbonate	5.13	2
Water	10.47	45

Several interesting features are evident in this assemblage of microwave spectra relative to the 2.45 GHz frequency at which the heating experiments were conducted. Both activated carbons show a much stronger absorption at 1.46 GHz and at frequencies between 1.80-1.85 GHz. LiOH absorbs more strongly in the 1.85-1.9 GHz region. A relatively broad band of enhanced susceptibility is seen for Molecular Sieve 13X between 1.825-1.910 GHz. The differences in transmission spectra between the wet and dry silica gel is particularly pronounced at 1.5, 1.75, and 1.81-1.87 GHz. By contrast, the presence of water results in little or no difference in silica gel spectra at 1.725, and between 1.95-2.6 GHz.

The above is only a cursory interpretation of the vast body of information contained within the reflection, transmission, and phase lag spectra. Unfortunately, the time constraints of the Phase I performance period did not permit a more thorough and thoughtful analysis.

IV. CONCLUSIONS.

Feasibility of the use of microwave heating for the fast and efficient thermal regeneration of sorbents for the removal of carbon dioxide, water vapor, and trace organics from contaminated air streams has been conclusively demonstrated. Microwave powered thermal regeneration of single sorbents and composite sorbent beds loaded with acetone, trichloroethylene (TCE), carbon dioxide, and water vapor has been achieved using a rectangular waveguide based test apparatus emitting at a frequency of 2.45 GHz. Both activated carbon and Carbosieve S-III were identified as excellent microwave regenerable sorbents for use in the removal of airborne organics. Water loaded silica gel, Molecular Sieve 13X, and Molecular Sieve 5A were also effectively regenerated under microwave irradiation at this frequency. Molecular Sieve 5A and a carbogenic molecular sieve prepared at NASA's Jet Propulsion Laboratory were identified as viable microwave regenerable CO_2 sorbents. A sorbent bed containing multiple media was challenged with air containing 0.5% CO_2, 300 ppm acetone, 50 ppm TCE, and saturated with water vapor. The composite bed was shown to effectively purify the contaminated air stream and to be completely regenerated by microwave induced heating.

Spectral studies of the reflection, transmission and phase shifts of microwaves irradiating a variety of sorbents over the frequency range between 1.37 - 2.6 GHz have indicated that significant differences in the dielectric loss characteristics (which are responsible for a material's susceptibility to microwave heating) occur between sorbents as a function of frequency. Frequencies have been identified with potential for more effective microwave heating of specific sorbents. Based upon these results, further development of this highly promising technology is highly recommended.

Currently, expendable cartridges containing activated carbon and lithium hydroxide are used for the removal of trace contaminants, CO_2, and water vapor inside the suit during EVA. A regenerable system is in the final stages of development for deployment during assembly of the future International Space Station *Alpha* (ISSA). This system will use metal oxide CO_2 sorbents in conjunction with other thermally regenerable media. Regeneration of these devices will take place within the cabin of the shuttle or space station and will operate using conventional resistive heating elements and will rely on conduction and convection of heat to the thermally regenerable media.

Based upon the successful microwave powered regenerations of sorbents for water vapor, carbon dioxide, and trace organics separately and in combination, it is highly probable that a more efficient and more convenient regenerable air purification system for EVA can be designed based upon this technology. For example, rather than removing expended sorbents from the EMU for regeneration, microwave power could be delivered directly to the EMU via coaxial cable, allowing regeneration to take place *in situ*. This would provide substantial savings in crew time. Additionally, significant improvements in size, weight, power consumption, regeneration efficiency, and regeneration times can be gained.

In addition to the obvious applicability to EVA and Advanced Life Support, two specific systems with strong potential for commercial application have also been identified.

These are the acetone-Carbosieve S-III and TCE-ZSM-5 combinations. The first system represents an environmentally benign method for the recovery of waste solvents in a variety of industrial chemical processes. Using the highly selective carbon based molecular sieve, and the extremely rapid thermal desorption capabilities inherent to microwave heating, acetone (or similar solvents) can be recovered from waste gas streams by sorption and then concentrated by flash thermal desorption for collection by condensation. The second commercial application exploits the fact that ZSM-5 is not only a sorbent for removal of airborne trichloroethylene, it is also an effective catalyst for the deep oxidation of this contaminant, particularly in the chromium form. Thus, the TCE-ZSM-5 system forms the basis for a combined environmental remediation process to achieve both the separation and the ultimate destruction of TCE.

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